FINAL REMOVAL DESIGN/REMOVAL ACTION WORKPLAN

MASTER METALS, INC. SITE

Cleveland, Ohio

PREPARED BY:

ENTACT

MARCH, 2002

ENTACT
environmental tactics in waste management

1360 North Wood Dale Road
Suite A
Wood Dale, Illinois
60191

April 9, 2002

Ms. Gwen Massenberg
U.S. Environmental Protection Agency
Remedial Response Branch
77 W. Jackson Blvd. SR-6J
Chicago, IL 60604-3590

Re: Final RD/RA Workplan and Final Design for the Master Metals Cleveland Site, Cleveland, Ohio

Dear Gwen,

Enclosed are two copies of the Final RD/RA Workplan and Final Design document for the Master Metals Site, Cleveland, Ohio. The reports have been revised to incorporate USEPA, OEPA and NOLCO comments on the Draft RD/RA Workplan and Pre-Final Design document and drawings.

Please feel free to call me at (630) 616-2100 if you have any questions.

Sincerely,

Patricia Vojack

Patricia Vojack, P.G.
ENTACT & Associates LLC

Cc: Ms. Sheila Abraham, Ph.D, OEPA – 2 copies
Mr. Terry Casey, Efficasey Environmental LLC – 1 copy
Mr. Charles Bredt, NOLCO - 1 copy

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FINAL REMOVAL DESIGN/REMOVAL ACTION WORKPLAN

FOR
THE MASTER METALS, INC. SITE
CLEVELAND, OHIO

PREPARED BY
ENTACT & Associates LLC.

March 2002

1.0 INTRODUCTION

1.1 Purpose and Objectives

In accordance with the objectives set forth in the Administrative Order of Consent (AOC) and the modified Statement of Work (SOW) for the Master Metal Inc. (MMI) Site in Cleveland, Ohio, ENTACT & Associates LLC (ENTACT) has developed this Removal Design (RD) and Removal Action (RA) Workplan to outline the procedures and methodologies to be used for the remedial action at the Master Metals Site. The objective of this RD/RA Workplan is to provide for the safe and efficient completion of the removal action pursuant to the Comprehensive Environmental Reponse. Compensation, and Liability Act of 1980, 42 USC §9601 (CERCLA), as amended by the Superfund Amendments and Reauthorization Act of 1986, Pub. L No. 99-499, 100 Stat. 1613 (1986) (SARA).

This RD/RA Workplan includes a comprehensive description of the work to be performed, and a schedule for both the completion of each major activity and submission of each deliverable. This plan consists of six sections, summarized below:

- **Section 1: Introduction** – Section 1 provides a description of the Site, including the location and history.
- **Section 2: Project Organization and Management** – Section 2 provides a description of the project team, project organization, and responsibilities.
- **Section 3: Scope of Work Tasks** – Section 3 includes a description of the main three primary SOW tasks, including project plans, RD phases, and RA construction.
- **Section 4: Removal Action and Construction** – Section 4 describes the major construction activities that will be implemented during the RA pursuant to the AOC and the SOW.
- **Section 5: Work Products and Reports** – Section 5 describes and lists the reporting requirements during the implementation and at the completion of the RA.
- **Section 6: Project Schedule** – Section 6 presents the project schedule which includes a schedule of completion for each required major activity and submission of each major deliverable.

1.2 Site Location and Description

The MMI Superfund Site (the "Site') covered under the AOC includes the former MMI lead facility (the "Facility") located at 2850 West Third Street, Cleveland, Cuyahoga County, Ohio and stockpiled, treated soils removed from the residential property at 1157, 1159 and 1167 Holmden Avenue (the "Holmden Properties") where lead-impacted material from Master Metals was deposited as fill (USEPA, 1999). The Site is situated in Township 7 North, Range 12 West, Section 17, ¼ NE, ¼ SW, ¼ SW, with coordinates obtained from the Facility Index System (FINDS) listed as 41 degrees, 28 minutes, 26 seconds latitude and -81 degrees, 40 minutes, 31 seconds longitude. The site location is illustrated in Figure 1-1.

The MMI property is a triangular-shaped parcel encompassing approximately 4.3 acres in the "flats" area of downtown Cleveland, a heavily industrialized sector of the city. The site is bordered on west by rail yards owned by the Baltimore & Ohio (B&O) Railroad, the east by West Third Street and B&O railroad tracks, and on the south by a dead-end road and an abandoned industrial property. LTV Steel owns the property to the south and north. The Cuyahoga River is located approximately 1,250 feet east of the facility and flows north toward Lake Erie (ENTACT,

1999). An athletic field and playground are situated approximately 1,000 feet to the west. The nearest residential property to the former facility is approximately 2,000 feet to the northwest (USEPA, 1999).

Major site features, prior to a 1997-1998 time-critical removal (TCR) action, included an office building, a secondary lead smelting furnace building, two large brick baghouses, the roundhouse building, storage buildings, material storage bins and boxes, and an above-ground storage tank farm (ENTACT, 1998). All buildings, except for the roundhouse and the attached office building in the northern corner of the property, were razed as part of the Phase I TCR (ENTACT, 1998). All remaining feedstock and debris materials were decontaminated and/or treated and disposed of off-site as either special waste or as hazardous waste (ENTACT, 1998). The MMI facility property is currently vacant with the exception of the roundhouse, and the majority of the open land surface is covered with concrete or asphalt except along the site boundaries. Current site features are illustrated in Figure 1-2.

Stormwater drainage is directed toward one of five on-site stormwater catch basins that connect to the combined sewer system operated by the Northeast Ohio Regional Sewer District (NEORSD) (ESC, 1991).

Topographic maps suggest that the direction of groundwater flow and surface water flow in the vicinity of MMI is to the northeast toward the Cuyahoga River (ENTACT, 1999).

1.3 SITE HISTORY

1.3.1 MMI Facility

The facility was constructed in 1932 on slag fill by National Lead Industries, Inc. (NL Industries) who owned and operated the facility as a secondary lead smelter, producing lead alloys from lead-bearing dross and scrap materials. NL Industries also engaged in battery cracking operations at this facility. In 1979, the facility was purchased from NL Industries by MMI who continued to run secondary lead smelter operations (USEPA, 2001a).

As part of their operations, the MMI facility received lead-bearing materials classified and regulated under Resource Conservation and Recovery Act (RCRA) as D008 hazardous waste from off-site sources (USEPA, 2001a). This waste was converted into lead ingots using pot and rotary furnaces equipped with baghouses to collect particulate matter from the furnace that consisted predominantly of lead dust. The material that accumulated in the furnaces/baghouses after smelting was classified as K069 hazardous waste. Finished lead ingots were stored in a roundhouse at the north end of the property prior to shipment off-site.

Based on background information, the by-products produced from smelting operations included furnace flux, slag, dross, baghouse fines and furnace sludge (USEPA, 2001a). With the exception of slag, which was tested and disposed of off-site, most of the lead-bearing by-products were recycled back into the furnace. Cooling water used in the operations was diverted to a combined sewer system operated by the NEORSD (ESC, 1991).

On November 19, 1980, Master Metals filed a "Part A Permit" pursuant to the newly-promulgated RCRA regulations, and obtained "interim status" under RCRA to operate specific waste piles and treatment units, as well as a container-based storage area for the hazardous lead-

bearing materials. On January 11, 1982, Master Metals filed for Chapter 11 bankruptcy through the U.S. Bankruptcy Court for the Northern District of Ohio but subsequently went into reorganization and operations at the facility continued. Though Master Metals had submitted a Part B RCRA permit application prior to November 8, 1985, on that date the facility lost interim status for the hazardous lead-bearing waste piles at the facility for failure to comply with financial requirements of 40 CFR Part 265, Subpart H.

On June 15, 1987, a complaint of RCRA violations was filed by the United States, seeking closure of the D008 and K069 waste piles at the facility. In response to this action, MMI presented a partial closure plan that included procedures to close these waste piles (USEPA, 2001a). Soil and groundwater sampling was conducted by MMI as part of this closure plan. Analytical data from the soils showed lead and cadmium present in the soils at concentrations below the then-applicable Environmental Profile (EP) toxicity criteria. Groundwater samples collected approximately ten feet below ground surface showed the presence of relatively low levels of lead and cadmium at levels just above the Ohio groundwater standards (ESC, 1991).

On January 15, 1990, Master Metals entered into Consent Decree with the United States to resolve RCRA continuing violations. In April 1990, MMI submitted to the USEPA a revised RCRA Part B Permit application for closure of various solid waste management units (SWMUs) on the facility (USEPA, 2001a).

Violations relating to noncompliance and poor operating practices are documented in various state and federal agency reports. These findings are summarized in the Section III of the AOC, presented in Appendix A. In January of 1992, the Ohio Environmental Protection Agency (OEPA) installed three ambient air monitors near the facility property. Quarterly air sampling results from the station immediately downwind of the facility showed repeated exceedences of the Clean Air Act's 42 USC National Ambient Air Quality Standard (NAAQ) for lead. MMI installed a sprinkling system in July 1992 in an attempt to prevent air-borne migration of the dust from the facility (USEPA, 2001a) but exceedences of the NAAQ for lead continued to be measured downwind of the facility. On September 9, 1992, MMI conducted a thorough cleaning of the facility in another attempt to minimize the effects of wind-blown facility dust.

On August 5, 1993, as a result of continuing RCRA violations, the OEPA Director ordered MMI to cease operating the facility until it could demonstrate compliance (USEPA, 2001a). Operations never did resume at the MMI facility and Bank One of Ohio took possession of all MMI cash collateral and accounts receivable. The current property owner remains MMI. The former facility president, Mr. Douglas Mickey, is deceased (USEPA, 2001a).

Following shutdown, MMI and the USEPA continued negotiations to resolve RCRA noncompliance issues. On March 28, 1995 the USEPA RCRA Division deferred the MMI Site to CERCLA for cleanup. On August 22, 1995, MMI withdrew all permits still in effect regarding its operation terminating its ability to legally treat, store or dispose of hazardous waste at the facility (USEPA, 2001a). Fifty-three potentially responsible parties (PRP Respondent Group) signed an Administrative Order by Consent (AOC) for the MMI facility that became effective April 17, 1997. The Order required the PRPs to conduct a Phase I TCR action and a Phase II Engineering Evaluation and Cost Analysis (EE/CA) for a non-time critical removal action for the facility pursuant to the National Contingency Plan (NCP) and the Superfund Accelerated Cleanup Model (SACM) guidance.

In accordance with the April 17, 1997 AOC Docket No: V-W-97-C both the Phase I TCR and Phase II EE/CA were completed by ENTACT on behalf of the PRP Respondent Group, as described in Section 1.4 of this Workplan.

An environmental evaluation of potential impacts associated with implementation of an excavation remedy was performed as part of the Proposed Plan (USEPA, 1999). The evaluation determined that most of the adverse effects associated with excavating soils would be short-term in nature and could be controlled by using good construction practices.

1.3.2 Holmden Properties

The Holmden Properties encompass approximately one-half of an acre and are located in a residential neighborhood, atop a hillside overlooking the flats. They are surrounded on the north, east and west by continuing residential areas and on the south and southeast by industrial areas located at the bottom of the hillside (USEPA, 2001a). In the late summer of 1987, lead-bearing material from MMI facility was allegedly deposited at the Holmden Properties as fill.

In 1991, the occupants of 1157 Holmden Avenue at the Holmden Properties contacted the OEPA to relate their concerns with the Master Metals fill material. In response, the OEPA collected soil samples at the Holmden Properties and found elevated concentrations of lead and cadmium. Based on the analytical results, OEPA required MMI to remove contaminated soils from the Holmden Properties. Following removal, the OEPA conducted a second soil sampling investigation in March, 1992 on the Holmden Properties and found additional lead-impacted soils. In December 1992, MMI removed additional soils from the Holmden Properties and conducted soil sampling following removal. The analytical results showed elevated levels of lead remained in the soils. The occupants of 1157 Holmden not able to return to their home, which was vandalized and later damaged by arson. The City of Cleveland condemned the house on August 18, 1995 and demolished it on February 22, 1996. Beginning on April 9, 1997, Ecology & Environment Technical Assistance Team (TAT) conducted an additional site investigation at the Holmden Properties and the results indicated between 2,000 to 3,000 cubic yards of lead-impacted material exceeding the 400 mg/kg default residential cleanup criteria was present.

On October 23, 1997, six potentially responsible parties (PRPs) signed an AOC for the Holmden Properties agreeing to conduct a TCR pursuant to the National Contingency Plan (NCP) and Superfund Accelerated Cleanup Model (SACM) guidance. On behalf of the Holmden Respondents, ENTACT conducted a time-critical removal action between November 10, 1997 and December 6, 1997 to remove contaminated soils exceeding a residential cleanup level of 400 mg/Kg along Holmden Avenue (ENTACT, 1998c). The excavated contaminated soils were stabilized to below a Toxicity Characteristic Leaching Procedure (TCLP) level of 0.75 mg/L, well below the Land Disposal Restriction (LDR) criteria, then stockpiled on the facility property (ENTACT, 1998c). Following excavation and confirmatory sampling to verify that the cleanup objective of 400 mg/Kg had been met, the Holmden Properties were restored to their original condition including revegetation (ENTACT, 1998c).

1.4 PREVIOUS REMOVAL ACTIONS

1.4.1 Phase I Time Critical Removal Action

The Phase I TCR was conducted by ENTACT on behalf of the PRP Respondent Group between June 9, 1997 through January 6, 1998 in accordance with the AOC Docket No: V-W-97-C. The TCR included the excavation, demolition, consolidation and/or removal of highly contaminated buildings, structures, soils, loose waste materials, industrial debris and other equipment to reduce the spread of, or direct contact with, documented contamination. This included the characterization and removal of non-hazardous materials, and removal, treatment, as necessary, and disposal of hazardous materials. The complete results of the TCR investigation are detailed in the Time Critical Removal Action Phase I Final Report, dated April 24, 1998 (ENTACT, 1998a).

Decontamination and/or demolition of the existing structures were conducted as part of the Phase I TCR scope of work. All materials deemed non-hazardous or recyclable were decontaminated prior to leaving the site. With the exception of the roundhouse and attached office building, all site structures were razed in accordance with the AOC.

All on-site surface areas not covered with concrete were excavated to a maximum depth of two feet or until historic slag fill materials (i.e., slag, cinders, etc.) were encountered. Lead concentrations in the remaining historic slag fill material were documented up to 39,000 parts per million (ppm) (ENTACT, 1998a). The excavated soils were stabilized to render the material nonhazardous and transported off-site to an approved Subtitle D landfill. Approximately 4,300 tons of treated soils were removed as part of this action (ENTACT, 1998a). Following excavation, the excavated areas were backfilled with clean fill material in accordance to the approved Phase I TCR Workplan.

As part of the Phase I TCR, approximately 4,800 cubic yards (yd^3) of solid non-hazardous waste, 500 y^3 of brick/concrete special waste, 21 tons of asbestos-containing materials, 1,160 y^3 of K069, D006, and D008 hazardous waste, 3,600 pounds (lbs.) of chromium trioxide, and over 200 bottles of laboratory chemicals were removed from the facility and properly disposed of in accordance with applicable state and federal guidelines. Approximately 3,000 gallons of liquid waste associated with drummed liquids in the roundhouse, six above-ground storage tanks and below grade sumps and catch basins were collected, characterized and disposed of off-site (ENTACT, 1998a). The site was also secured with fencing and signs to prevent unauthorized entry (ENTACT, 1998a).

1.4.2 Phase II Engineering Evaluation and Cost Analysis

The Phase II EE/CA was conducted by ENTACT on behalf of the PRP Respondent Group to develop an appropriate cleanup objective or Risk-Based Remediation Goal (RBRG) for the residual concentrations of lead remaining in soils at the MMI Site (ENTACT, 1998b). In accordance to AOC Section V.2, the EE/CA included the following five tasks:

1. Generation of EE/CA Workplan;
2. Generation of EE/CA Support Sampling Plan;
3. Completion of Support Sampling;
4. Generation of the EE/CA Data Report; and
5. Generation of the EE./CA Report.

Complete results of the EE/CA investigation are included in the EE/CA Report dated November 23, 1998 (ENTACT, 1998b).

Historical analytical data collected at the site between 1990 and 1998 were evaluated to determine the nature and extent of contamination at the site related to former site activities, and to identify where additional investigation was required to complete delineation of the facility-associated impacts. The historic slag fill, which pre-dates and underlies the facility as well the majority of the surrounding area, contains elevated lead concentrations that are not related to former facility operations and therefore, not included in the removal actions for this site. Based on this review, additional soil and groundwater sampling were conducted to complete characterization of the nature and extent of lead contamination related to former facility operations. The EE/CA characterization investigation included on-site and off-site soil sampling, a perimeter XRF lead survey on surface soils, and groundwater sampling.

The on-site soil sampling included the advancement of seven borings within the facility perimeter. Results indicated that five of the seven borings exceeded 1,500 mg/Kg lead at total depth. Historic slag was encountered at approximately three to four feet which is consistent with the information collected during the Phase I TCR (ENTACT, 1998b). The on-site sampling indicated that significant lead concentrations, up to 35,000 mg/Kg, remained in on-site soils to a depth of three to four feet below grade. These areas were either covered with the existing concrete surface or had been excavated and backfilled with two feet of clean fill as part of the Phase I TCR. Therefore in areas where the concrete was competent and in uncovered areas that were excavated as part of the Phase I TCR, the potential for further entrainment of airborne lead had been mitigated and was no longer considered a concern (ENTACT, 1998b). However a potential for airborne lead releases did exist in areas where the concrete was compromised. These areas were recommended for repair to mitigate this airborne migration route (ENTACT, 1998b)

A perimeter surface soil survey was conducted adjacent to the fence line along the western, eastern and southern boundaries of the MMI facility property using an X-Ray Fluorescence (XRF) instrument, at nineteen locations designated in Figure 1-3. Results of the perimeter lead survey showed lead levels ranging from 931 ppm to 36,587 ppm within the upper 12 to 24 inches of soils, decreasing rapidly with depth. The surficial elevated lead levels currently pose a potential ingestion or inhalation threat, and were recommended for further remedial action (ENTACT, 1998b).

Off-site sampling included the collection of nine off-site surface soil samples along Quigley Avenue. The results showed the average lead concentration to be below the Superfund residential soil screening level of 400 mg/Kg, indicating that any potential airborne lead impacts from the former MMI facility are minimal. No further action was recommended (ENTACT, 1998b).

Groundwater sampling conducted in 1991 showed total lead concentrations ranging from 0.45 mg/L to 1.35 mg/L, total chromium concentrations ranging from 0.02 mg/L to 1.33 mg/L, and lesser concentrations of arsenic and cadmium (CTI, 1991). Groundwater sampling of the three existing monitoring wells during the 1998 EE/CA investigation showed the presence of lead, arsenic, cadmium and chromium at levels that have either remained at, or have declined from, the 1991 sampling results. Groundwater is not used as a source of drinking water within a four-mile radius of the site, with Lake Erie supplying the greater Cleveland area with its drinking water supply. Based on the low concentrations of metals in the groundwater and the lack of any potential downgradient receptors, the groundwater migration pathway was eliminated as a concern (ENTACT, 1998b).

The EE/CA assessment verified that lead was the predominant hazardous constituent of concern at the site, with lesser occurrences of arsenic. Removal action directed at lead exceedences

would also address the co-located elevated levels of arsenic. Based on a streamlined risk evaluation, a RBRG for lead of 1,000 mg/Kg was established for on-site and off-site perimeter soils (ENTACT, 1998b).

An environmental evaluation of potential impacts associated with implementation of an excavation remedy was performed as part of the Proposed Plan (USEPA, 1999). The evaluation determined that most of the adverse effects associated with excavating soils would be short-term in nature and could be controlled by using good construction practices.

1.5 Administrative Order of Consent

Based on the findings of the Phase II EE/CA, an Administrative Order of Consent (AOC) was entered into between the USEPA and the PRP Respondent Group in Spring 2002 to perform a non-critical removal action, as described in the Statement of Work (SOW) to address remaining lead impacts at the site that are associated with former facility operations. The AOC is presented in Appendix A.

The SOW includes the following tasks:

- Clear and grub areas requiring excavation of all trees and brush for disposal off-site.
- Demolish applicable above-grade concrete and metal structures remaining on-site after the Phase I TCR demolition activities as detailed in the design specifications. Sized concrete construction debris will either be used as a sub-base material in areas to be covered with the asphalt cover or will be transported off-site disposal as construction debris. All wood, bricks or metal debris that are removed will be disposed of off-site as construction debris.
- Establish a coordinate grid system along the perimeter of the property outside the fence line and in on-property areas where excavation is required.
- Excavate off-property soils along the western, eastern and southern perimeter of the MMI facility, that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first. XRF screening technology will be used to guide the depth of the excavations during removal.
- Excavate designated on-property soils that are not under concrete or the proposed asphalt cover (including grids I1, J1 and K1 excavated during the Phase I TCR) that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first.
- Conduct confirmatory soil sampling from the excavation floor in grids where the excavation was terminated prior to reaching the historic slag fill material to confirm that all soils (other than historic slag) that are above the cleanup level have been excavated and removed.
- Backfill all excavated areas once verified to have met the RBRG or have reached historic slag fill, and grading to promote positive drainage in accordance with the design documents. Backfill for areas not covered by asphalt or concrete will be filled with clean imported fill material that has been approved for use based on analytical results and is suitable to maintain vegetative growth.

- Stabilize excavated soils to meet the applicable LDRs for contaminated soils for lead, and any underlying hazardous constituent (UHC) during waste profiling, to render the material nonhazardous for either use as fill in low areas beneath the proposed asphalt cover or for off-site disposal at an approved Subtitle D facility.

- Conduct verification sampling of treated soils using TCLP lead analysis to verify the material has been rendered non-hazardous for lead prior to either placement in low areas beneath the proposed asphalt cover or for off-site disposal as nonhazardous waste.

- Off-site disposal of all treated soils not placed beneath the proposed asphalt cover, in accordance with the SOW and the approved design plan.

- Place an asphalt cover over the deteriorated area of the concrete located in southern portion of the site in accordance with the design documents.

- Recondition existing concrete surfaces not under the asphalt cover by sealing any significant cracks and breaks that extend through the concrete surface, followed by encapsulation of the concrete surface, in accordance with the approved design plan.

- Abandon of all existing monitoring wells on site in accordance to applicable State of Ohio regulations (OAC-3745-9-10).

- Remove any existing solid waste including Investigative Derived Waste (IDW) from previous or current removal actions.

- Install a perimeter chain-link fence and three double-swing gates at the completion of the RA to control site access at the site in accordance with the design documents.

- Development of an Operation and Maintenance (O&M) Plan to ensure the integrity of the remedy by maintaining and repairing the concrete and asphalt cover, and the perimeter fencing for a period of thirty (30) years, and as specified in the AOC.

2.0 PROJECT ORGANIZATION AND MANAGEMENT

Project organization, responsibilities, lines of communication, and reporting procedures are described in the following sections.

2.1 PROJECT ORGANIZATIONAL CHART

Figure 2-1 illustrates the lines of authority of the Project Management Team for overseeing and implementing the required remedial action (RA) at the MMI Site in Cleveland, Ohio. ENTACT's assigned management team may change during implementation of the RA. If there is a change in personnel of ENTACT's management team, the modification will be communicated to US EPA's RPM and the Project Coordinator. Qualifications and experience of ENTACT's Management Team is provided in Appendix D, Quality Assurance Project Plan, in Attachment QAPP-D.

2.2 MANAGEMENT RESPONSIBILITIES

USEPA CERCLA Remedial Project Manager, Gwen Massenberg

The USEPA CERCLA Remedial Project Manager has the overall responsibility for all phases of the Remedial Action Workplan.

Project Coordinator, Terry Casey, Efficasey Environmental LLC.

The Project Coordinator's prime responsibility will be to ensure proper coordination among various project stockholders. These stakeholders include the USEPA, OEPA, City of Cleveland, NOLTCO, Bredt & Zanick, LLC, the Project Manager, and the Respondents to the Order.

ENTACT Project Manager, Mike Stoub, ENTACT

The ENTACT Project manager will be responsible for ensuring that the site activities are implemented and completed in accordance with the AOC, SOW, the U.S. EPA-approved RD/RAA Workplan and federal, state, and local regulations. He will be responsible for the following tasks:

- Providing personnel and equipment for remedial activities;
- Provide the Project Coordinator and U.S. EPA's RPM the names and qualifications, if appropriate, of the contracted laboratory, disposal facilities, and transporters used to implement the RA;
- Ensuring that ENTACT's associates perform their designated duties in strict accordance with the Health and Safety Plan;
- Ensuring required quality assurance/quality control (QA/QC) procedures are properly implemented and documented;
- Notifying appropriate personnel identified in the Health and Safety Plan (HASP) in the event that the Contingency Plan is implemented;
- Ensuring the RA is completed consistent with the approved schedule;
- Facilitating effective communications between the Project Coordinator and U.S. EPA's RPM;

- Ensure that all documents and reports that ENTACT is required to generate meet the requirements of the approved workplan;
- Communicate any request for modifications to the approved workplan to the Project Coordinator and U.S. EPA; and,
- Promptly notifying the Project Coordinator and U.S. EPA's RPM in the event of unforeseen field conditions and/or problems are encountered.

Corporate Health and Safety Officer, Jonathan Patlak, ENTACT & Associates LLC

The Corporate Health and Safety Officer will coordinate and provide oversight for the Health and safety issues at the site. He will be responsible for conducting the Health and Safety Orientation Meeting before the RA is implemented. He will review weekly health and safety updates from the site and conduct several inspections at the site during the RA.

Regulatory/Technical Leads, Pat Vojack P.G., Mark Waxali, P.E., ENTACT & Associates LLC

Ms. Vojack will provide regulatory and technical support to the Project Manager in ensuring that the site activities are implemented and completed in accordance with the AOC, SOW, the U.S. EPA-approved RA Workplan and federal, state, and local regulations. Mr. Waxali will provide engineering expertise and direction in implementation of the design documents and in ensuring construction activities conform to the approved design documents. The regulatory and technical leads will provide technical support to the ENTACT Project Manager in the areas of wastewater management and treatment, solid and hazardous waste management, air monitoring, and any other technical design requirements for the RA.

Management Control Process

The Project Coordinator has responsibility for successfully implementing the requirements of the AOC. The ENTACT Project Manager has overall responsibility for successfully completing the remedial action at the site. This includes safely completing technical Statement of Work items, fulfilling contractual obligations, compliance with the approved workplan, and meeting or exceeding the established project schedule and budget. The Project Manager will accomplish these objectives by monitoring the work progress, reviewing and planning each project task with experienced technical staff and the Field Project Manager, and ensuring the appropriate and sufficient resources are available to the Field Project Manager and the On-Site QA/QC Officer.

The Project Manager will receive daily progress reports from site personnel appraising him of the status of planned, ongoing, and completed work, including QA/QC performance and health and safety, site-specific issues. In addition, the Project Manager will be apprised of any potential problems and recommendations for solutions and/or corrective action.

2.3 QUALITY ASSURANCE RESPONSIBILITIES

US EPA Region 5 Superfund's Quality Assurance Reviewer, Richard Byvik

U.S. EPA Superfund Quality Assurance Coordinator has the responsibility to review and approve all Quality Assurance Project Plans. In addition, the U.S. EPA Quality Assurance Coordinator is responsible for conducting external performance and system audits of the laboratory and evaluating analytical field and laboratory procedures.

ENTACT Quality Assurance/Quality Control Manager, Patricia Vojack, P.G., ENTACT & Associates LLC

Ms. Vojack will be responsible for setting up the QA Program for this site and ensuring that all approved QA/QC procedures for this project are being followed. In addition, the ENTACT QA/QC Manager will be responsible for ensuring that data validation is completed for 25 percent of the sample results from the analytical laboratory by an outside Chemist.

On-Site QC Officer, ENTACT, Inc.

The on-site Quality Control Officers will be responsible for performing required quality control testing at the site. The on-site QC officer will operate independently of ENTACT's Project Manager and Field Project Manager. The QC Officer will communicate any QA/QC issues related to the site to the QA/QC Manager. The QC officer will have the authority to correct and implement additional measures to assure compliance with the approved workplan, including the QAPP. Specific responsibilities will include:

- Adhere to the approved QAPP;
- Document any deviations to the plan with a justification for the deviations, and if necessary appropriate notification in accordance with the approved workplan;
- Secure necessary sampling tools, bottles, packaging/shipping supplies, chain-of custody documents, etc. in accordance with the approve workplan;
- Collect or direct the collection and ship samples at the frequencies and for laboratory analysis parameters specified in the QAPP;
- Document the location, time, and date of all samples that are collected and shipped to the laboratory;
- Interface with the superintendents such that the sample collection is coordinated with the general progression of the work;
- Notify the Project Manager, and the U.S. EPA of any sampling activities associated with the implementation of the approved workplan; and
- Obtain analytical results and report the data to the Project Manager and U.S. EPA's RPM.

3.0 SCOPE OF WORK TASKS

3.1 INTRODUCTION AND OUTLINE OF TASKS

In order to expedite the Removal Action and facilitate redevelopment of the property, the seven tasks outlined in Section 3 of the SOW have been consolidated into four tasks as described below. The requirements listed in the SOW as Task 1 (Removal Design Workplan), Task 3 (Removal Action Workplan), Task 4 (Workplan Addendum) and Task 7 (Performance Monitoring) in the SOW have been combined into a single task, Task 1, Development of Draft and Final RD/RA Workplan. The Removal Design Phases outlined in Task 2 have been streamlined into the submittal of Task 2, Pre-Final Design and Final Design Document. The consolidation of the submittals proceeded with approval from the USEPA, in accordance to Section III of the SOW.

The required schedule for these submittals is presented in Figure 3-1. The consolidated tasks are as follows:

Task 1: Development of the RD/RA Workplan, including the following, required supporting plans:

- Performance Standard Verification Plan
- Field Sampling and Analysis Plan
- Quality Assurance Project Plan
- Contingency Plan
- Treatability Study Report
- Erosion Control Plan
- Community Relations Plan
- Health and Safety Plan and Contingency Plan (Separate Attachment)

Task 2: Development of the pre-final and final design documents for the RD/RA Workplan

- Pre-final Design document which includes the draft Construction Specifications with all associated drawings, and the Construction Quality Assurance Project Plan (CQAPP) for the implementation of the RA.
- Final Design that includes the final Construction Specifications and CQAPP, construction estimates, and construction schedule for the implementation of the RA.

Task 3: Implementation of Removal Action Construction

- Pre-construction inspection meeting
- Mobilization
- Storm water control measures
- Treatment staging area and on-site treatment containment area construction
- Air monitoring
- Dust suppression/engineering controls
- Clearance of subsurface utilities and other obstructions
- Site security
- Establishment of grid coordinate system
- Demolition of existing above-grade concrete and metal structures in footprint of asphalt cover

system
- Clearing of trees
- Soil excavation, stockpiling and treatment
- Post-excavation confirmatory soil sampling
- Backfilling and site restoration
- Off-site disposal of treated material
- Transportation and disposal
- Cleanup and demobilization
- Pre-final Inspection Meeting and Report
- Final Inspection Meeting
- Completion of Removal Action Report
- Completion of Work Report

Task 4: Operation and maintenance

3.2 TASK 1 – WORKPLAN AND SUPPORTING PLANS

Task 1 consists of the preparation of the RD/RA Workplan and supporting plans for submittal to the U.S. EPA. The following sections describe the contents of each of the supporting plans, which includes the Performance Standard Verification Plan (PSVP), Field Sampling and Analysis Plan (FSAP), Quality Assurance Project Plan (QAPP), Treatability Study Report (TSR), Erosion Control Plan (ECP), Community Relations Plan (CRP), and the Health and Safety Plan (HASP) and Contingency Plan (CP).

3.2.1 Performance Standard Verification Plan (PSVP)

The PSVP summarizes all performance standards to be met during and after the RA. The PSVP includes, among other criteria, the methodologies for treatment and for conducting TCLP testing. The FSAP, HASP, and the QAPP support the PSVP.

The PSVP is presented in Appendix B of this RD/RA Workplan.

3.2.2 Field Sampling and Analysis Plan (FSAP)

The FSAP supplements the QAPP and addresses sample collection activities, including confirmatory soil sampling, XRF screening, sampling for treatment and disposal, and air sampling.

The FSAP is presented in Appendix C of this RD/RA Workplan.

3.2.3 Quality Assurance Project Plan (QAPP)

The Quality Assurance Project Plan (QAPP) is a site-specific plan for sample analysis and data handling. It includes a description of sample custody control, field and laboratory quality control checks, and corrective actions.

The QAPP is presented in Appendix D of this RD/RA Workplan.

3.2.4 Treatability Study Report (TSR)

The TSR presents the results of the laboratory treatment studies ENTACT has conducted on lead-impacted material obtained from the site. The treatability study for site soils was performed in 1997 as part of the Phase I activities. The purpose of the study was to determine the on-site treatability of representative soils. The plan also includes a brief discussion of ENTACT's patented treatment system and patented treatment additives that would be used in the on-site treatment process.

The TSR is presented in Appendix E of this RD/RA Workplan.

3.2.5 Erosion Control Plan (ECP)

The ECP contains a description of the remedial construction site conditions, hazardous materials and handling, erosion controls, and best management practices that will be implemented to minimize the potential for contaminated run-off from the site. The plan meets the substantive requirements of OEPA's erosion and sediment control requirements for construction activities.

The ECP is presented in Appendix F of this RD/RA Workplan.

3.2.6 Community Relations Plan (CRP)

The CRP describes the methods that will be used to inform the surrounding community of the planned remedial activities at the site. In addition, the CRP identifies an easily accessible repository for information about the remedial actions to be implemented at the site.

The CRP is presented in Appendix G of this RD/RA Workplan.

3.2.7 Health and Safety Plan (HASP)

The site-specific HASP describes all procedures and criteria to protect on-site personnel and area residents from physical, chemical, and all other hazards potentially posed during the implementation of the RA. The HASP includes detailed descriptions of levels of protection, personal protective equipment, decontamination procedures, and contingency procedures.

The HASP is presented under separate cover and accompanies this RD/RA Workplan

3.2.8 Contingency Plan (CP)

The CP describes procedures to be used in the event of an accident or emergency at the MMI Site, including corrective action measures that will be taken if there is an exceedence of performance standards required for air at or from the site. The plan also includes a brief discussion of the process to be followed if an emergency or accident occurs at the site during the RA. Responses to emergencies or accidents are described in more detail in the site-specific Health and Safety Plan.

The CP is included with the HASP (under separate cover) and accompanies this RD/RA Workplan.

3.3 TASK 2 – DESIGN PHASES

Task 2 of the SOW includes the Pre-final and Final Design documents, the Draft/Final CQAPP, construction estimates, the planned trucking route, and the final project schedule for the construction and implementation of the RA. The Pre-Final Design represents a 95 percent complete design, including reproducible drawings and specifications suitable to implement the RA. Upon the U.S. EPA's approval of Final Design construction estimates, final CQAPP, and final Project Schedule, the Pre-Final Design will serve as the Final Design. The Pre-Final Design submittals include the draft CQAPP, the truck route and the draft Project Schedule.

3.3.1 Construction Quality Assurance Project Plan (CQAPP)

The Construction Quality Assurance Project Plan (CQAPP) describes the quality assurance program to ensure that the completed project meets or exceeds all design criteria, plans, and specifications. The CQAPP includes protocols for sampling and testing to monitor construction activities and reporting requirements, such as summary status reports and inspection data sheets. The CQAPP is presented in the draft Pre-Final Design document accompanying this RD/RA Workplan.

3.3.2 Project Schedule

The project schedule presents the estimated time frames to complete the major components of the RA. A more detailed project schedule will be submitted with the draft Pre-Final Design document.

3.3.3 Truck Route

The Final design will present the planned truck route for transporting impacted soils to the treatment staging area and the transporting of stabilized material to the approved off-site disposal facility.

3.4 TASK 3 - REMOVAL ACTION AND CONSTRUCTION

In accordance with the schedule in Section IV of the SOW, the RA will be implemented as described in the RD/RA Workplan and Design Document. The removal action and construction activities and associated documentation and reports are described in detail in Section 4.0 of this Workplan.

3.5 TASK 4 - OPERATION AND MAINTENANCE

Task 4 consists of the preparation of the Draft/Final Operation and Maintenance Plan to cover maintenance and repair of the existing concrete and asphalt cover and perimeter fencing for a period of 30 years. The O&M Plan will include all elements listed under Section III, Task 6 of the SOW. A Draft O&M Plan will be submitted concurrently with the Final Design document and the Final O&M Plan will be submitted no later than the final Pre-Inspection meeting, in accordance with the schedule provided in the SOW.

4.0 REMEDIAL ACTION AND CONSTRUCTION

4.1 PRE-CONSTRUCTION INSPECTION AND MEETING

The Respondents and ENTACT will meet with the U.S. EPA and OEPA for a pre-construction inspection and meeting at the MMI Site. The purpose of the meeting will be to:

- Review methods for documenting and reporting inspection data;
- Review methods for distributing and storing documents and reports;
- Review work area security and safety protocols;
- Discuss any appropriate modifications of the draft CQAPP to ensure that site-specific considerations are addressed; and
- Conduct a site walk-around to verify that the design criteria, plans, and specifications are understood and to review material and equipment storage locations.

The pre-construction inspection and meeting will be documented by one of the ENTACT attendees and the transcribed minutes will be transmitted to all parties.

4.2 MOBILIZATION AND SITE PREPARATION

Project mobilization and site preparation activities will be conducted to prepare the site for full-scale removal activities. Achieving a quality project according to schedule requires experienced planning and organization during the mobilization phase of the project. The site preparation activities listed below will be conducted for the MMI Site Project:

- Notify appropriate agencies for emergency response in accordance with the Health and Safety and Contingency Plan;
- Notify suppliers and vendors to allow for timely and efficient project start up;
- Site survey and photo documentation to verify condition of remaining site structures that are adjacent to excavation areas, and overhead obstructions;
- Location of the construction office and connection of electricity, water, telephone and facsimile;
- Establish treatment area and haul road according to the approved truck route;
- Establish the coordinate grid system with 50-foot by 50-foot grid cells;
- Schedule a site utility line location (gas, electric, telephone fiber and wire, storm and sanitary sewer, water and cable);
- Construct barricade safety fences;
- Identify personnel and equipment access areas;
- Identify and construct material storage and loading areas;
- Construct decontamination areas for personnel and equipment;
- Establish work and exclusion zones;
- Install storm-water/erosion controls;
- Install water management systems for collection, dust suppression, and discharge;
- Install and initiate air monitoring systems for site perimeter, work areas and personnel; and
- Setup a meteorological data collection center in the administrative trailer.

Figure 4-1 illustrates the general site layout including approximate location of the excavation areas, treatment area, asphalt cover area and the following work areas (exclusion zone, contamination reduction or decontamination zone and support zone).

In order to prepare for efficient excavation, soil stabilization and loading operations, ENTACT will align numerous aspects of site control, including:

- Establish site inspection protocol and documentation requirements;
- Secure the impacted work areas to control site entry and exit.
- Implement ENTACT's sign-in log to document entry of visitors and personnel on site; and
- Post the appropriate signage to restrict and control site access.

Work Zones will be established around the perimeter of the facility. Tape and signs will be installed to identify the Exclusion, Contamination Reduction, and Decontamination Zones. Level C Personal Protective Equipment (PPE) will be required to enter the Exclusion Zone. Access to the zones will be controlled.

4.2.1 Stormwater/Erosion Control Measures

Stormwater and erosion control measures will be implemented before the management of material is initiated at the site. These control measures will include the use of berms, hay bales or drainage channels to prevent off-site run-off and control overland flow as described in the Erosion Control Plan in Appendix F to the RD/RA Workplan. These measures will be implemented, maintained, and removed pursuant to the requirements of the approved RD/RA Workplan.

4.2.2 Staging and On-site Treatment Areas

A staging area for treated material will be constructed in conjunction with the layout described in Figure 4-1. The staging area will be located on the level concrete surface. Staged soil piles will be temporarily covered with polyurethane sheeting at the end of the day's activities or prior to inclement weather to minimize the generation of leachate or airborne lead. A containment berm will be constructed around the perimeter of the staging area to prevent any surface water run-off and provide a means of collecting any water that may leach through the stockpiled material. An on-site borrow source or the treated stockpile soils from the Holmden Properties removal will be used for the berm material if possible. The staging area may be moved during the project to increase efficiency of operations. Collected wastewater will be used for dust suppression in areas requiring excavation or on stockpiles awaiting treatment, as needed. Wastewater not used for dust suppression will be analyzed for the NEORSD discharge parameters to determine if the water can be discharged to the municipal sewer system, pending approval from the City of Cleveland.

The on-site treatment of the excavated soils will be conducted according to the Treatability Study Report, presented in Appendix E of this RD/RA Workplan. The Phase IV land disposal restrictions applicable to contaminated soils, for lead and any potential underlying hazardous constituent (UHC), were met for soils that contain a hazardous waste. The Treatability Study was designed to meet the applicable LDR requirement for any material that, when generated, exhibited a hazardous characteristic. The alternative LDR treatment standards for hazardous lead-contaminated soils is 7.5 mg/L TCLP lead, but the soils will be treated to be below the

hazardous characteristic criterion for lead of 5.0 mg/L to render the material nonhazardous. The frequency of the sampling of the treated material to be either placed in low areas beneath the asphalt cover or to be disposed of off-site as nonhazardous waste is described in Section 3.3 of the FSAP. A detailed discussion of the treatability study is presented in Appendix E of this RD/RA Workplan

Near completion of the on-site treatment of the material, the berm material surrounding the staging area will be tested for TCLP lead, treated if necessary to render the material nonhazardous for either on-site placement beneath the asphalt cover or off-site disposal. The concrete pad underlying the treatment containment area will be decontaminated after all treatment activities are completed.

4.2.3 Air Monitoring

Air monitoring will be conducted during the project to determine the concentrations of air-borne lead to ensure that all work personnel and surrounding residents are not exposed to levels of lead in excess of the regulated limits, and to ensure that contaminants are not migrating off site. For this project, Clean Air Act monitoring methodologies will be employed to monitor for respirable dust and lead emissions in addition to the OSHA defined air monitoring for the following purposes:

- Health and safety;
- Monitor dust suppression effectiveness; and,
- Monitor dust borne lead concentrations.

Plans in the RD/RA Workplan that describe air monitoring are the FSAP (Appendix C) and the HASP. The FSAP includes procedures for air sampling using total suspended particulate (TSP) samplers and area/personal air monitors in Section 4.0 of the FSAP. Air-sampling procedures for personal air monitoring in Section 7.1.

4.2.4 Dust Suppression/Engineering Controls

Site preparations will include positioning and implementing dust suppression and engineering control measures to ensure that air emissions are maintained at "no visible emissions" at the MMI Site boundary/fence line during the construction phase of the RA. To control dust, ENTACT will employ misting using high-pressure, low-volume, portable water spray units in the excavation, staging, treatment and loading areas and along site roads.

4.2.5 Subsurface Utilities and Other Obstructions

Prior to beginning heavy equipment operations, ENTACT will file utility line locate requests with locating services for underground utilities. Existing overhead power lines that prevent remedial activities will be either relocated or removed. Caution and awareness of power lines that remain in place will be emphasized in site safety meetings.

4.2.6 Site Security

Access to the MMI Site will be controlled by the existing perimeter fence and gates, as well as by the project manager. Site visitors that enter the work zone will be required to read, sign and

comply with the HASP and must wear the appropriate personal protective equipment before entering work areas. All visitors will be required to sign the logbook, located inside the ENTACT administrative office trailer.

4.2.7 Establishment of Coordinate Grid System

A coordinate grid system (CGS) will be established in order to provide a coordinate system for tracking sampling and excavation activity in the field. Figure 4-1 depicts the approximate location of the CGS. The CGS will employ square grids of 50 feet by 50 feet superimposed over the existing site and perimeter area extending out beyond the fenceline. This coordinate system will be used to provide benchmark locations and reference markers for 1) excavation documentation, 2) XRF field-screening activities, and 3) post-excavation confirmatory soil sampling. Installation and use of the CGS is described in the FSAP (Appendix C of this RD/RA Workplan.).

4.3 CLEARING OF SITE

The excavation areas will be cleared of all trees and grubbed to grade for proper drainage using standard construction equipment. The trees shall be cleared to as near ground level as practicable and disposed of off-site.

Existing concrete structures that are present within the footprint of the asphalt cover system will be demolished and the debris disposed of as construction debris at an approved landfill.

In addition, all on-site drums left behind by previous contractors will be opened and the contents properly disposed. The empty drums will then be decontaminated using a steam-cleaner or pressure washer for possible recycling or disposal along with the construction debris from demolition of existing concrete structures (i.e. remaining walls from precious demolition efforts, loading docks and storage areas). Waste material generated during clearing and removal activities (i.e. PPE, concrete debris, etc.) will be disposed of off-site at an approved landfill. Any material designated for recycling will be transported to the designated decontamination area and steam-cleaned or pressure-washed to remove any surface lead before leaving the site.

4.4 DEMOLITION OF CONCRETE STRUCTURES

The existing concrete structures, including partial walls, stalls and other above-grade structures, excluding the dock area, will be demolished to grade using conventional construction equipment in accordance to the design specifications. All demolition services will be performed in accordance with applicable federal, state, and local requirements.

The concrete debris may be used as subbase material in areas where an asphalt cover is to be placed or disposed of off-site as construction debris. All other debris will be stockpiled for off-site disposal as construction debris.

4.5 EXCAVATION, CONSOLIDATION, AND/OR TREATMENT OF SOILS

On-property soils not covered with concrete or the cover system, and off-site perimeter soils along the eastern, western and southern boundaries of the property will be excavated until either the RBRG of 1,000 mg/Kg is reached or until historic slag fill is encountered (the "risk goals"),

whichever comes first. On-property areas excavated and backfilled with clean fill in accordance to the approved Workplan during the Phase I TCR will not be addressed unless identified in the SOW as requiring additional removal. Identified grids requiring re-excavation will include only those grids that will not be covered with an asphalt cover.

Soils will be excavated with conventional construction equipment. The perimeter excavation will begin at the northeastern corner off Third Street and proceed in a southerly direction so as to minimize trafficking over areas where remedial action has occurred. For on-property soils addressed during the Phase I TCR but requiring re-excavation, the one-half to two feet of clean sand fill used as backfill will be removed and stockpiled for testing for the parameters described in Section 4.9 to determine if the material can be re-used as backfill in areas outside the asphalt cover. The stockpiled sand can placed without testing as fill in areas where an asphalt cover will be placed.

Excavation will be guided by the use of an XRF field screening instrument and will be terminated either when the performance criteria is achieved or the historic slag criteria is encountered. The XRF will analyze for total lead. For any grids where excavation is terminated prior to reaching the historic slag fill, the achievement of the performance criteria utilizing the XRF will be verified by the collection of a confirmatory sample for total lead analysis at the approved fixed laboratory verification. Once either the performance criteria has been confirmed to be met, or when historic slag fill is visually encountered, the grid will be considered successfully excavated and backfilled with clean fill material.

Excavated soils exceeding the 1,000 mg/Kg criteria will be consolidated and staged for treatment in the treatment staging area as illustrated in Figure 4-1. During treatment, the soils will be spread out in the treatment containment area and the pre-determined volume of additive required to achieve the applicable performance standard for soils (<5.0 mg/L TCLP lead) will be applied over the top the material. The additive and soils will then be thoroughly mixed using either a soil stabilizer or the bucket of the backhoe until a homogenous blend of soils and additive is achieved. The treated soils will then be staged in the post-treatment staging area for verification sampling prior to off-site disposal. A detailed discussion of the treatment system and additives used is provided in Appendix E of the RD/RA Workplan.

Following stabilization, the treated material will be stockpiled in 250 cubic yard piles for the first 1,000 cubic yards of material, then in 500 cubic yard piles thereafter and analyzed by TCLP lead testing to ensure the treatment standard has been met. Upon receipt of verification results, the material will either be used to fill low areas beneath the proposed asphalt cover or will be transported off-site to an approved Subtitle D landfill facility. Transportation and disposal for treated soils will be described in the Final Design document that will be submitted to U.S. EPA pursuant to Section III of the SOW and the approved RD/RA Workplan.

The volume of treated material to be transported off-site is estimated at approximately 3,100 cubic yards. This includes the existing stockpiled Holmden Avenue treated material, and the perimeter and on-site soils requiring excavation and treatment (1,800 cubic yards) based on the EE/CA sampling results (USEPA, 2001b; ENTACT, 1998b). All treated material placed for use in the cover system will be consolidated and graded to meet the requirements specified in the approved design document.

4.6 CONSTRUCTION OF ASPHALT COVER

The southern portion of the site designated in Figure 4-1, where the concrete is deteriorated, will be covered with asphalt in accordance with the design specifications. The asphalt cover will be designed to achieve the proper load-bearing capacity to permit industrial land reuse. Prior to installing the asphalt, all low areas and pits designated on the design drawings will be filled with stabilized material to grade. Any water that has accumulated in the pits will be pumped into temporary tanks for testing to determine whether or not the water can be discharged to the existing sewers once approval is obtained from the NEORSD. Test parameters will include those required by the NEORSD.

The asphalt cover will have a minimum thickness of 4-inches thick and include 2.75 inches of intermediate coarse aggregate layer and a 1.25 inch surface aggregate layer. A base course may be used as a subbase to bridge and fortify two adjacent areas where an elevational difference has been noted (i.e. areas bordered by concrete curbs that are proposed to be removed as part of demolition). Specifications on the design, construction, and applicable testing requirements for the asphalt cover at the MMI site will be presented in the Pre-Final Design Specifications and Construction Quality Assurance Plan (Plan).

The asphalt cover has been designed to provide an engineered barrier over the underlying existing fill that may contain lead-contaminated material, since lead is the primary contaminant of concern at the Site. The asphalt cover has also been designed to minimize the potential of a release from the site by providing containment that protects human health and the environment and prevent migration of the waste by air dispersion, surface water runoff, groundwater migration, or direct contact.

The asphalt layer will be sloped to promote direct site drainage to the existing sewer system in order to prevent site ponding for redevelopment purposes.

The detailed construction schedule will be presented in the Pre-Final Design Specifications and Construction Quality Assurance Plan (Plan). An estimated schedule is shown in Figure 6-1 of this Workplan.

4.7 REFURBISHMENT OF EXISTING CONCRETE SURFACE

The existing concrete layer that will remain on site outside of the asphalt cover will be inspected to ensure that the integrity of the concrete is intact for future land reuse. Areas with significant cracks or deterioration will be reconditioned by sealing with an impermeable epoxy or with concrete, followed by encapsulation of the concrete surface in accordance to the approved design plan.

4.8 MONITORING WELL ABANDONMENT

Four shallow monitoring wells were installed in the unconsolidated material beneath the MMI to a depth of 15 feet below grade (CTI, 1991). Only three of the four shallow monitoring wells were located during a well location survey performed as part of the EE/CA investigation in 1997. The locations of these remaining wells are illustrated in Figure 1-2.

In accordance with the SOW and AOC, all remaining wells and/or test borings at the site will be abandoned in accordance with Ohio Administrative Code (OAC) 3701-28-07 and the 1996 *State of Ohio Technical Guidance for Sealing Unused Wells*. Per Appendix 4 of the Ohio Technical guidance document, wells completed in unconsolidated formations may be satisfactorily sealed with neat cement or sodium bentonite.

Since the monitoring wells have been installed as flush mounts through the existing concrete cover, removal of the riser and screen will not be feasible. Instead, the wells will be backfilled with clean sand to one foot above the top of the screen (9 feet below grade). A one-foot layer of bentonite pellets will then be placed above the sand either through a tremie pipe and tamped down to ensure there is no bridging, and hydrated. The remaining annular space will be pressure-grouted on one continuous motion form the bottom up, using a tremie pipe. The casing will be cut off flush with the concrete surface, and the concrete surface repaired with epoxy or cement and encapsulated in accordance to the procedures discussed in Section 4.6. In accordance with Ohio Revised Code (ORC) 1521.05(B), a well sealing report will be filed with the Ohio Department of Natural Resources (ODNR) on forms supplied by the Department.

4.9 BACKFILLING AND SITE RESTORATION

ENTACT will backfill all excavation areas outside the asphalt cover, and all unpaved areas disturbed by construction, with clean, suitable fill, including a minimum of six inches of topsoil suitable for vegetative growth. The backfilled areas will be graded to promote positive drainage and to control any additional ponding of water that may occur during implementation of the remedy. All on-property catch basins have been determined to be functional.

In addition, as required under Section II, 1.1 of the SOW, the western portion of the site that was excavated during the Phase I TCR, will be re-graded will clean material and appropriately sloped to promote positive drainage.

Imported fill brought on-site from outside sources will be sampled to verify that the off-site fill materials are acceptable. The sampling will either be performed by ENTACT or by the supplier who will provide the necessary documentation that the material meets the OEPA criteria. If ENTACT performs the backfill characterization sampling, the sampling locations, methodologies, frequency of testing, and analytical protocols are described in FSAP.

Sample analyses required to determine whether the imported backfill material is acceptable include the eight RCRA metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver), volatile organic compounds (VOCs), pesticides/PCBs, and total petroleum hydrocarbons (TPH). Should the TPH value exceed the OEPA action level for TPH, the fill will be analyzed for semivolatile organic compounds (SVOCs).

Imported fill sampling will be performed at a rate of one grab sample for every 10,000 cubic yards of the same source material. Changes in the imported outside source location will require that the full parameter suite be repeated for that source material. The FSAP (Appendix C) and the QAPP (Appendix D) provide additional details on the sampling requirements and procedures for the backfill characterization.

4.10 TRANSPORTATION AND DISPOSAL OF EXCAVATED MATERIAL

The transportation and the disposal plan for any treated material not used to fill low areas beneath the asphalt, will presented in the truck route included as part of the Final Design Specifications.

4.11 CLEAN-UP AND DEMOBILIZATION

Upon completion of all site activities, all temporary construction facilities and utilities will be removed or disconnected. All trash, debris, and extra soil shall be removed from the site.

A Pre-final Inspection shall be conducted with representatives from the U.S. EPA, OEPA, ENTACT and the PRP Respondent Group. ENTACT will notify the USEPA within 30 days after a preliminary determination has been made that the construction is complete. The purpose of the inspection is to determine whether all aspects of the RD/RA Workplan and Design Plans have been implemented at the site, and whether the remedy is operational and meeting the Performance Standards.

5.0 WORK PRODUCTS

5.1 DAILY, WEEKLY AND MONTHLY REPORTS

ENTACT will prepare and maintain daily work reports and other records to summarize all site activities performed during completion of removal activities. At a minimum, the daily work reports will include a listing of personnel on-site, equipment utilized, work performed, problems encountered (if any) and resolutions, and related information.

ENTACT will prepare status reports on a weekly basis to summarize activities performed at the site during the previous week.

ENTACT will also prepare written monthly progress reports that:

- Describe the actions which have taken place during the month;
- Include a summary of all results of sampling and tests and all other data received or generated during the month;
- Identify all documents completed and submitted during the month;
- Describe all actions which are scheduled for the next six weeks, and information regarding construction progress;
- Include any Workplan modifications proposed/approved; and
- Describe activities undertaken in support of the community relations plan during the month and in the near future.

These monthly progress reports shall be submitted to USEPA and to the State by the tenth day of every month. ENTACT will notify USEPA of the occurrence of any change in schedule described in the monthly progress report for the performance of any activity no later than five days prior to performance of the activity.

An authorized representative of ENTACT will sign all reports (other than the monthly progress report described above).

5.2 EMERGENCY NOTIFICATION

Upon the occurrence of any event during the performance of the RA that ENTACT is required to report pursuant to CERCLA Section 103, 42 U.S.C. § 9603, or Section 304 of the Emergency Planning and the Community Right to-Know Act, 42 U.S.C. § 11004, ENTACT will notify the U.S. EPA within 24 hours of the onset of the event.

5.3 PHOTOGRAPHIC DOCUMENTATION

Photographs will be taken in order to serve as a pictorial record of work progress, problems, and mitigation activities. ENTACT's file at the site will contain color prints, labeled with the date and subject of the photograph. Photographic reporting data sheets, where used, will be cross-referenced with observation and testing data sheet(s), and/or construction problem and solution data sheet(s). Photographic documentation will be included in the RA Final Report.

5.4 REMEDIAL ACTION SUBMITTALS

During the implementation of the RA the following submittals will be provided to U.S. EPA and the OEPA for review and/or approval pursuant to the schedule established in the SOW and the approved RA Workplan:

- Final Design Document
- Draft Operation and Maintenance Plan
- Draft Phase II Groundwater Monitoring Program Plan
- Final Operation & Maintenance Plan

5.5 INSPECTION MEETINGS

During the implementation of the RA, at a minimum, the following meetings will be conducted at the site pursuant to the schedule in the approved SOW and the approved RA Workplan:

- Pre-Construction Inspection
- Pre-Final Inspection
- Final Inspection

5.6 FINAL INSPECTION AND RA REPORTS

Within 15 days after completion of the Pre-final inspection, ENTACT will submit the Pre-final Inspection Report. Within 45 days following a fully successful final inspection, ENTACT will submit a written report documenting remedial action activities and requesting certification.

6.0 PROJECT SCHEDULE

A project schedule for the required construction activities and the major deliverables is presented in Figure 6-1.

7.0 REFERENCES

Compliance Technologies, Inc. (CTI), January 17, 1991. *Groundwater Analyses Report for Master Metals, Inc., Cleveland, Ohio*

Ecology & Environment (E&E), August, 1992. *Site Assessment Report for the Master Metals, Inc. Site, Cleveland, Cuyahoga County, Ohio.* Prepared for Duane Heaton, Deputy Project Officer, Emergency Support Section, EPA Region 5.

Environmental Strategies Corporation (ESC), February 15, 1991. Environmental Risk Assessment Final Report, Master Metals Site, Cleveland, Ohio. Prepared for Master Metals, Inc..

ENTACT, April 24, 1998a. Phase I Final Report for Time-Critical Removal Action at the Master Metals Site, Cleveland, Ohio. Prepared for the EPA Region 5 on behalf of the Master Metals PRP Group in Response to the April 17, 1997 Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA.

ENTACT, Inc. (ENTACT), November 23, 1998b. Phase II Engineering Evaluation and Cost Analysis (EE/CA) Report for the Master Metals Site, Cleveland, Ohio. Prepared for the EPA Region 5 on behalf of the Master Metals PRP Group in Response to the April 17, 1997 Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA.

ENTACT, Inc. (ENTACT), February 6, 1998c. Final Report for Removal Activities at the Holmden Avenue Site, Cleveland, Ohio. Prepared for the EPA Region 5 on behalf of the Holmden Avenue PRP Respondent Group .

ENTACT, May 9, 1997. Phase I Time-Critical Removal Action Workplan for the Master Metals Site, Cleveland, Ohio. Prepared for the EPA Region 5 on behalf of the Master Metals PRP Group in Response to the April 17, 1997 Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA.

United States Environmental Protection Agency (USEPA), 2001a. *Administrative Order by Consent Pursuant to Section 106 of the Comprehensive Environmental Response, Compensation and Liability Act of 1980 –Master Metal Superfund Site, Cleveland, Ohio.*

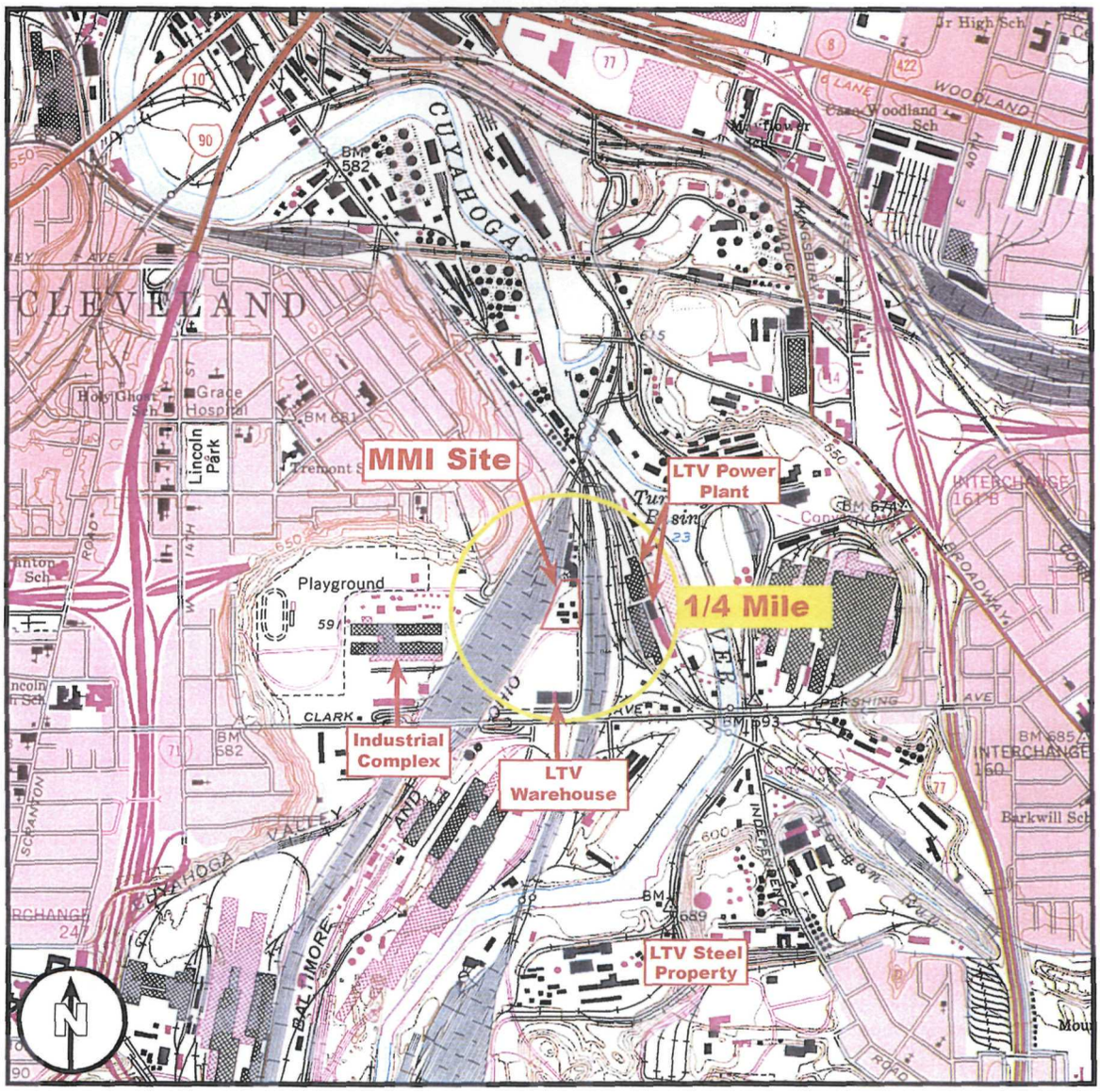
USEPA, 2001b. *Statement of Work (SOW) for the Design/Construction and Cleanup at the Master Metals Superfund Site, Cleveland, Cuyahoga County, Ohio.*

USEPA, March, 1999, U.S.EPA *Proposes Clean-up Plan for Master Metals Site, Cleveland, Ohio.* Office of Public Affairs, Region 5, Chicago, Illinois.

USEPA, April 17, 1997, *Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA. Docket No:*

SITE LOCATION MAP

CLEVELAND SOUTH QUADRANGLE OHIO-CUYAHOGA CO.
7.5 MINUTE SERIES (TOPOGRAPHIC)



SCALE 1:24 000

1 MILE
1 0
1000 0 1000 2000 3000 4000 5000 6000 7000 FEET
1 5 0 1 KILOMETER

CONTOUR INTERVAL 10 FEET
NATIONAL GEODETIC VERTICAL DATUM OF 1929
DEPTH CURVES AND SOUNDINGS IN FEET—DATUM IS LOW WATER 570.5 FEET

MN GN
6½° 116 MILS 0°27' 8 MILS

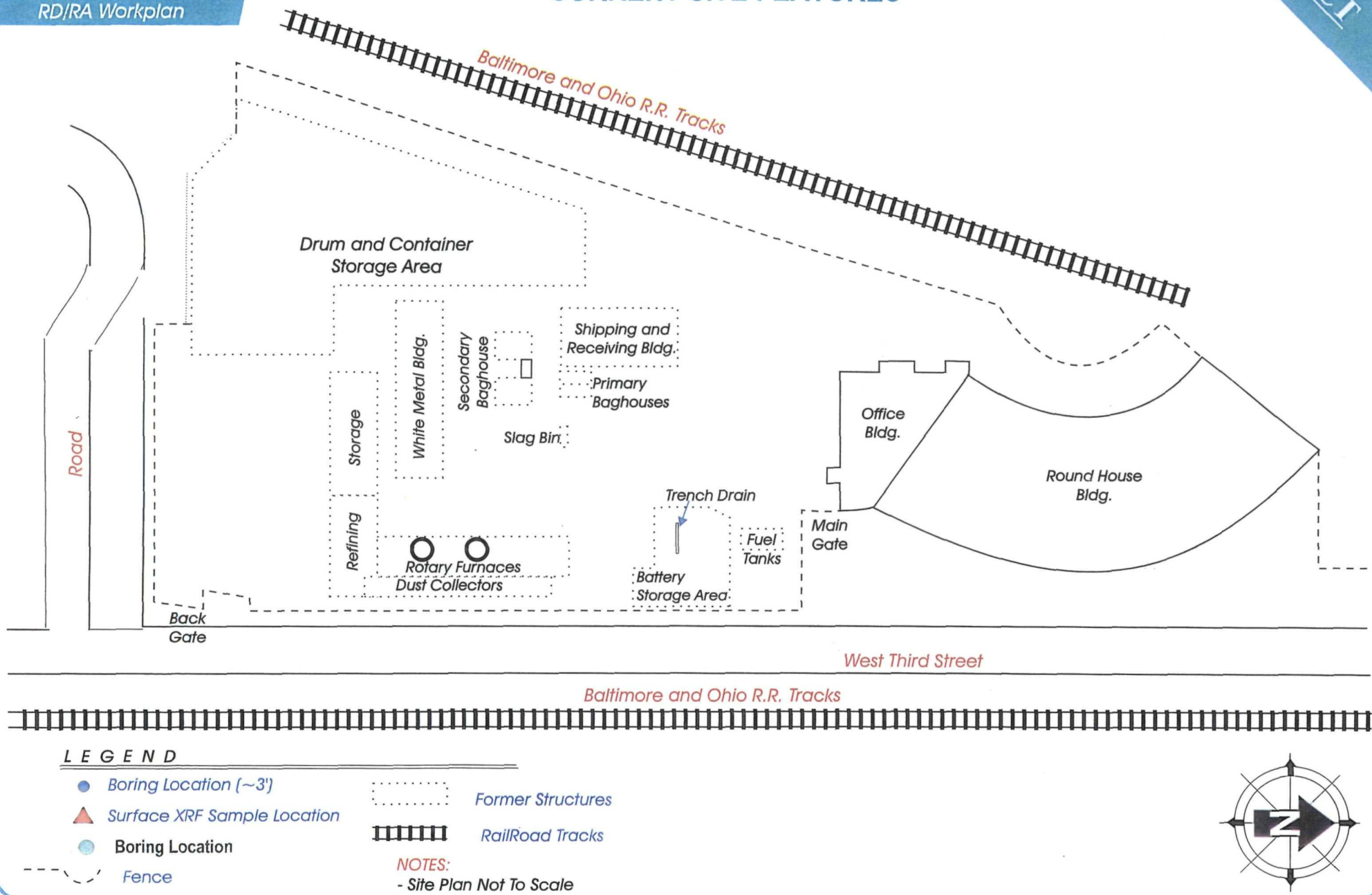
UTM GRID AND 1984 MAGNETIC NORTH DECLINATION AT CENTER OF SHEET

Figure 1-1

Master Metals
Cleveland, Ohio
RD/RA Workplan

FIGURE 1-2
CURRENT SITE FEATURES

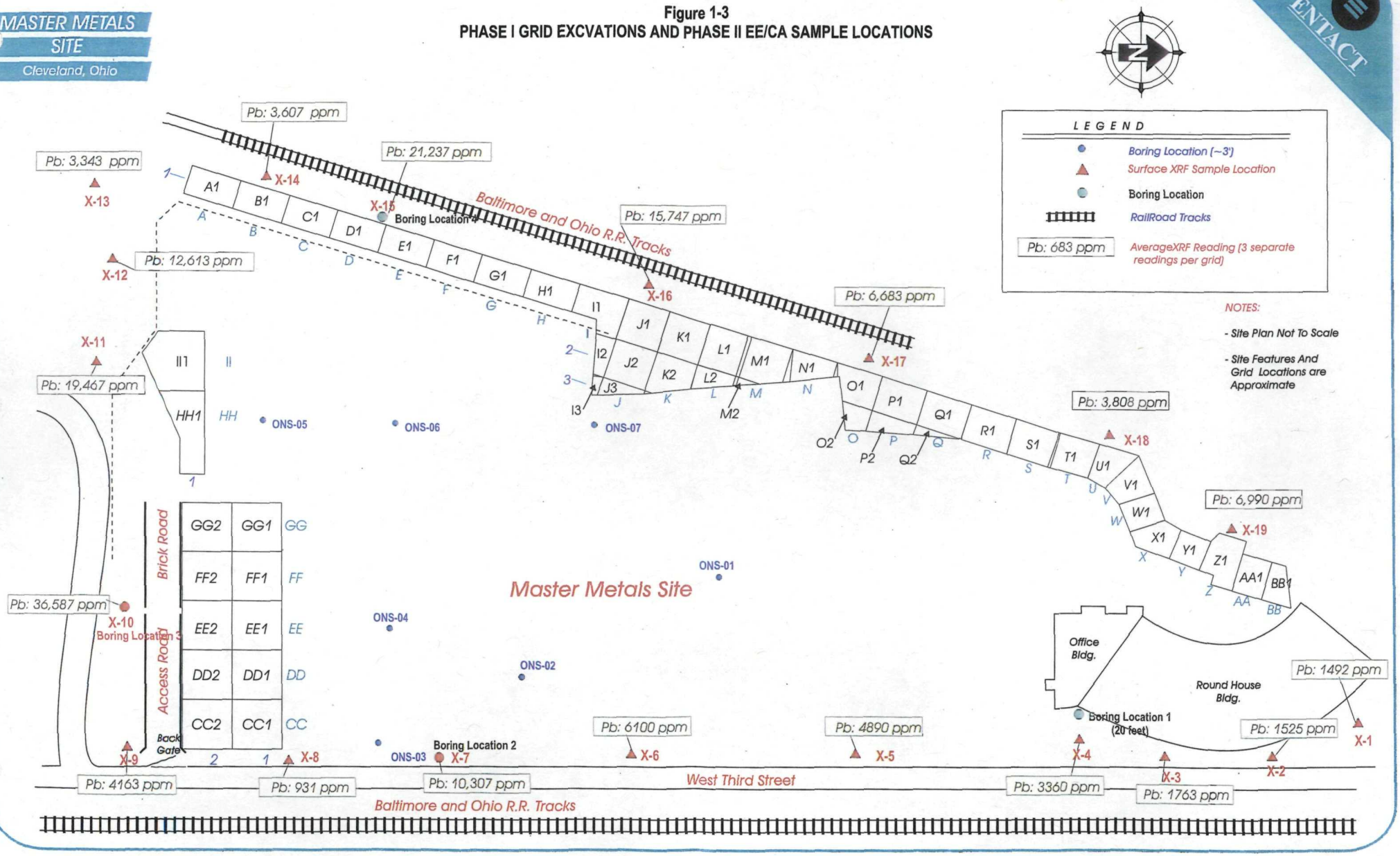
ENTACT



MASTER METALS SITE
Cleveland, Ohio

ENTACT

Figure 1-3
PHASE I GRID EXCVATIONS AND PHASE II EE/CA SAMPLE LOCATIONS



Master Metals
Cleveland, Ohio

ENTACT
September 26, 1997

Figure 2-1
PROJECT ORGANIZATIONAL CHART

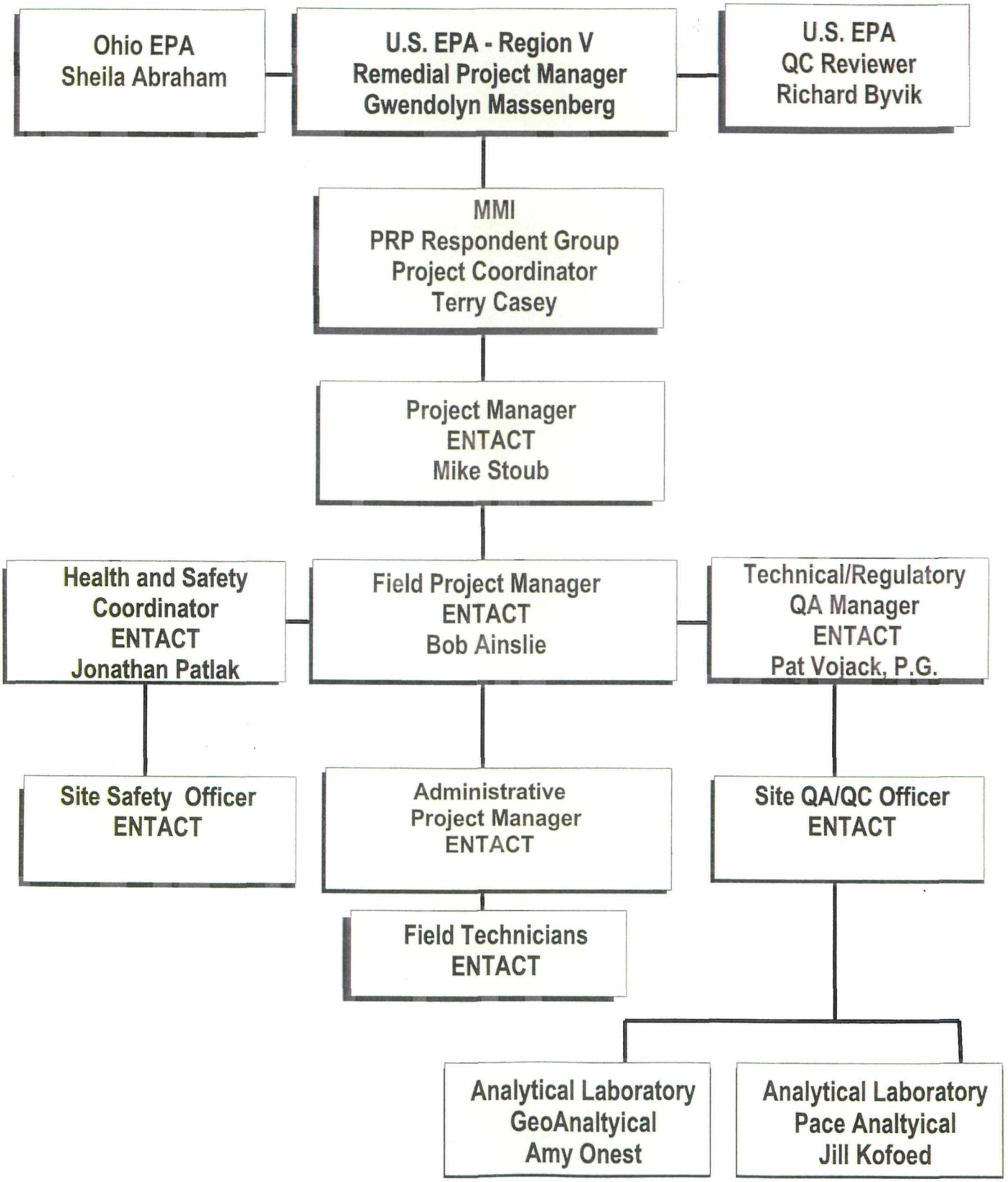


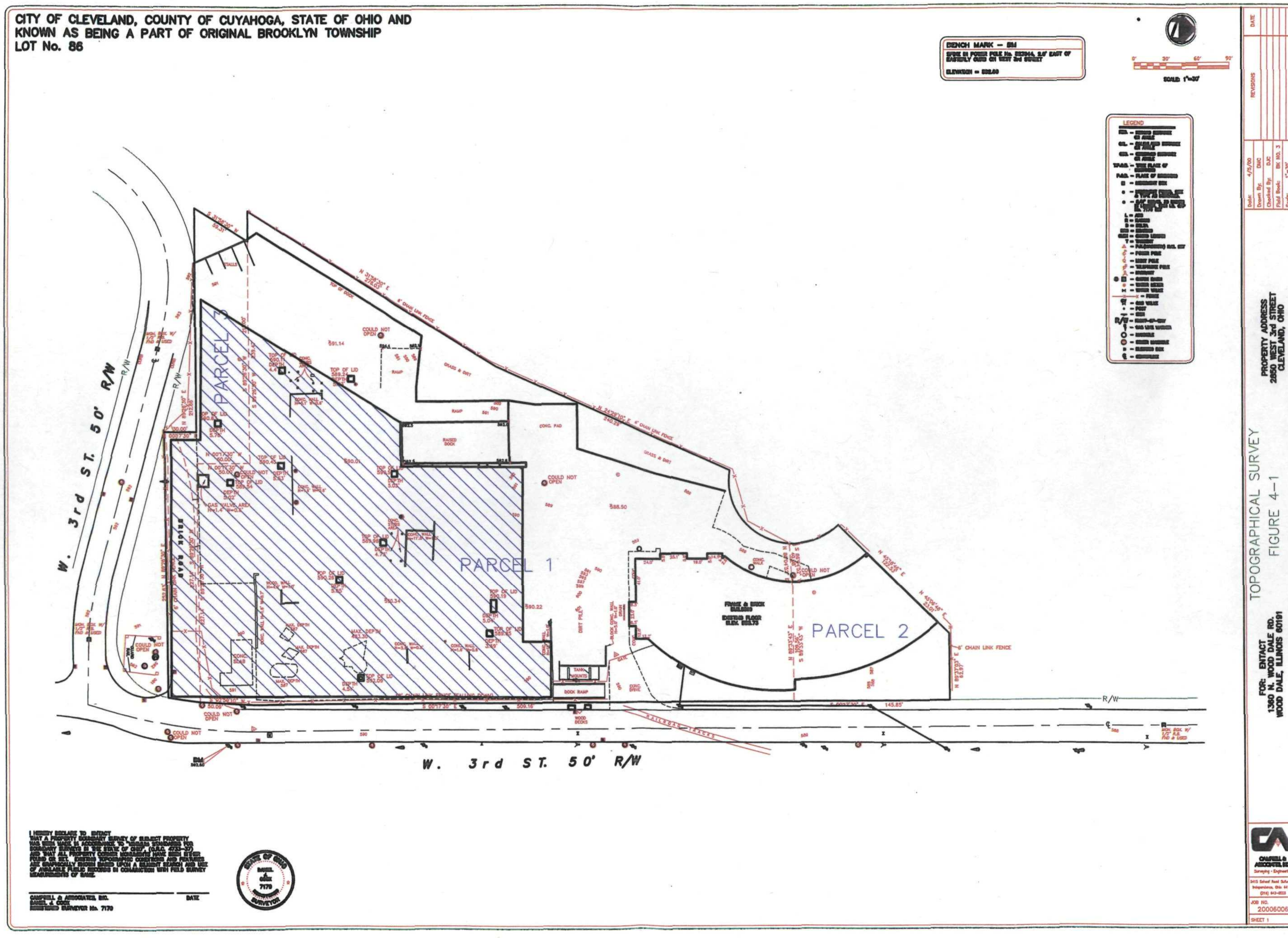
Figure 3-1
Deliverable Schedule
Master Metals, Inc., Cleveland, Ohio

Mandated Submittals / Meetings	Due Date to Agency
Removal Design Phase:	
Draft RD/RA Work Plan [1]	60 days after effective date of order
Final RD/RA Work Plan	30 days after receipt of comments on Draft
Pre-final Design [2]	60 days after effective date of order
Final Design/Draft O&M Plan	30 days after receipt of Agency comments on Pre-design
Removal Action Phase:	
Award of RA Contract Letter	30 days after receipt of Agency's Approval of RD/RA Workplan
Pre-construction inspection and meeting	15 days after award of RA Contract
Initiate Construction	15 days after pre-construction meeting and inspection
Pre-final Inspection	No later than 15 days after completion of construction
Pre-final Inspection Report	15 days after completion of pre-final inspection
Final Inspection	15 days after completion of work identified in prefinal inspection report
Final O&M Plan	No later than Pre-final Inspection
Completion of RA Report	due 45 days after fully successful final inspection
Completion of Work Report	due 45 days after completion of all remedial activities including O&M.
Monthly Progress Reports	Due on monthly basis throughout RD/RA following submittal of Draft WP

NOTES:

[1]: *Draft RD/RA Work Plan will include the following documents: Work Plan, Performance Standards Verification Plan, Field Sampling Plan, Quality Assurance Project Plan, Treatability Study, Erosion Control Plan, Community Relations Plan, and Health and Safety and Contingency Plan (submitted under separate cover).*

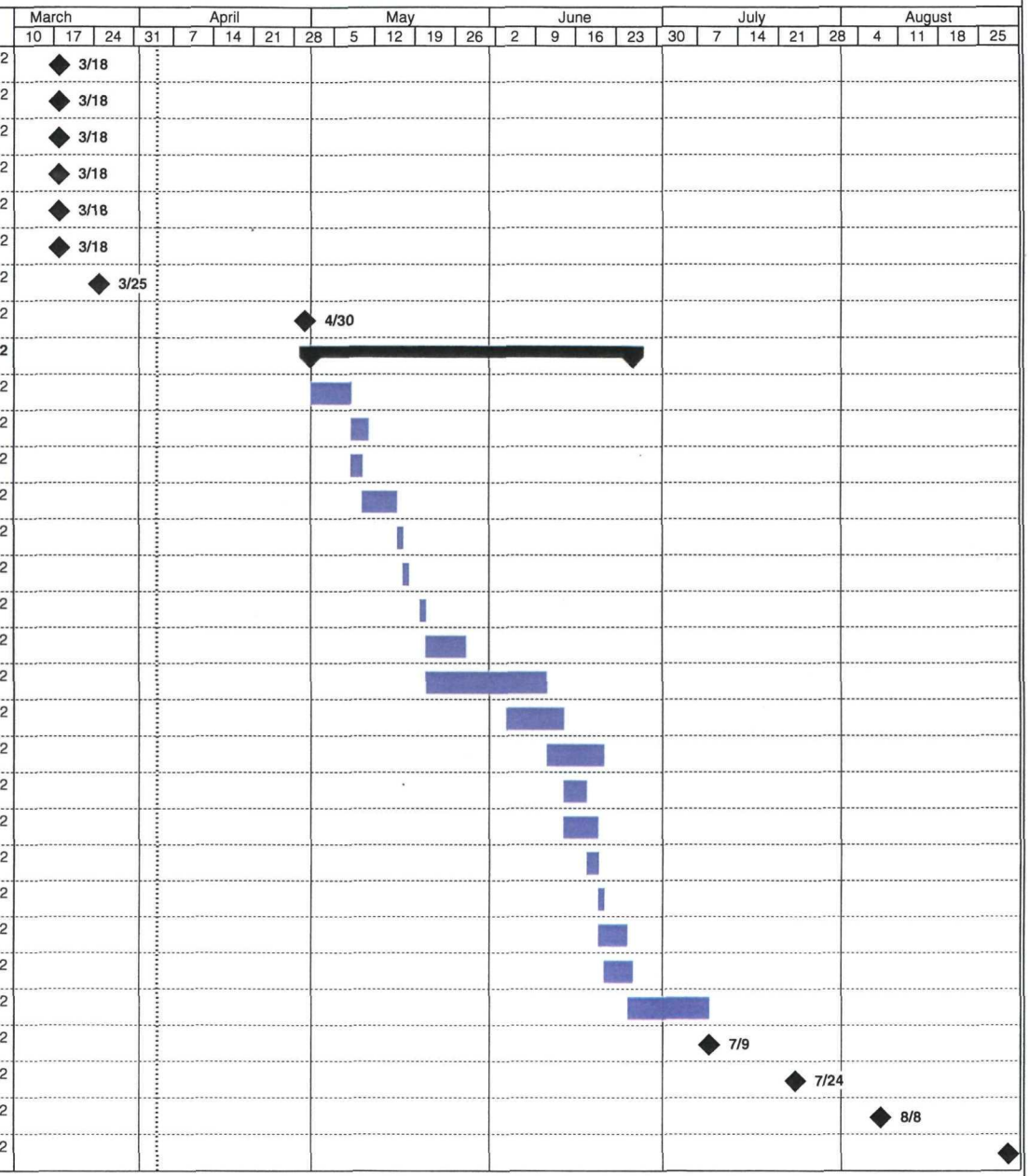
[2]: *95% complete design*



CITY OF CLEVELAND, COUNTY OF CUYAHOGA, STATE OF OHIO AND
KNOWN AS BEING A PART OF ORIGINAL BROOKLYN TOWNSHIP
LOT No. 86
BENCH MARK - BM
SCALE: 1"=30'
LEGEND
PARCEL 1
PARCEL 2
PARCEL 3
W. 3rd ST. 50' R/W
6' CHAIN LINK FENCE
COULD NOT OPEN
DOCK RAMP
R/W
TOPOGRAPHICAL SURVEY
FIGURE 4-1
PROPERTY ADDRESS
2850 WEST 3rd STREET
CLEVELAND, OHIO
FOR: ENTACT
1360 N. WOOD DALE RD.
WOOD DALE, ILLINOIS 60191
CAMPBELL & ASSOCIATES, INC.
Surveying • Engineering
JOB NO.
20006006
SHEET 1

Master Metals, I Cleveland, Ohio
Schedule for Remediation Activities
Thu 4/4/02

ID	Task Name	Duration	Start	Finish
2	Submittal of Final Workplan & Design (Complete)	0 days	Mon 3/18/02	Mon 3/18/02
3	Preparation of Work Plan and Design Documents (Complete)	0 days	Mon 3/18/02	Mon 3/18/02
4	Customer Review of Design Documents (Complete)	0 days	Mon 3/18/02	Mon 3/18/02
5	Incorporate comments on WP and Design Documents (Complete)	0 days	Mon 3/18/02	Mon 3/18/02
6	OEPA and USEPA Document Review (Complete)	0 days	Mon 3/18/02	Mon 3/18/02
7	Approval of Work Plan and Design Documents	0 days	Mon 3/18/02	Mon 3/18/02
1	AOC Signed by All Parties	0 days	Mon 3/25/02	Mon 3/25/02
8	Pre Construction Conference	1 day	Tue 4/30/02	Tue 4/30/02
9	**Field Activities**	**40 days**	**Wed 5/1/02**	**Tue 6/25/02**
10	Mobilization (Air Monit.;Erosion Ctrl; Stormwtr Ctrl)	5 days	Wed 5/1/02	Tue 5/7/02
11	Abandon Monitoring Wells	3 days	Wed 5/8/02	Fri 5/10/02
12	Clear and Grub	2 days	Wed 5/8/02	Thu 5/9/02
13	Abandon Underground Utilities	4 days	Fri 5/10/02	Wed 5/15/02
14	Clean & Dispose Drums & Contents	1 day	Thu 5/16/02	Thu 5/16/02
15	Remove Site Fence	1 day	Fri 5/17/02	Fri 5/17/02
16	Set-up Temporary Construction Fence	1 day	Mon 5/20/02	Mon 5/20/02
17	Demolish Structures/Dispose Offsite	5 days	Tue 5/21/02	Mon 5/27/02
18	Excavate and Stabilize Perimeter Soils	15 days	Tue 5/21/02	Mon 6/10/02
20	Offsite Disposal	8 days	Tue 6/4/02	Thu 6/13/02
19	Backfill, Topsoil, Seed Excavated Perimeter Areas	8 days	Tue 6/11/02	Thu 6/20/02
22	Place asphalt	2 days	Fri 6/14/02	Mon 6/17/02
23	Refurbish Concrete	4 days	Fri 6/14/02	Wed 6/19/02
24	Install New Perimeter Fence	2 days	Tue 6/18/02	Wed 6/19/02
25	Survey	1 day	Thu 6/20/02	Thu 6/20/02
26	Demobilization	3 days	Thu 6/20/02	Mon 6/24/02
21	Fill and Grade Low Areas, Topsoil, Seed	3 days	Fri 6/21/02	Tue 6/25/02
27	Preparation of O&M Plan	10 days	Tue 6/25/02	Mon 7/8/02
28	Pre-Final inspection	1 day	Tue 7/9/02	Tue 7/9/02
29	Pre-Final Inspection Report	1 day	Wed 7/24/02	Wed 7/24/02
30	Final Inspection	1 day	Thu 8/8/02	Thu 8/8/02
31	Final Report	1 day	Fri 8/30/02	Fri 8/30/02



APPENDIX A

STATEMENT OF WORK (SOW) AND USEPA LETTER REVISIONS TO SOW

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5

IN THE MATTER OF:	)	**Docket No.**
	)	
Master Metals, Inc.,	)	**ADMINISTRATIVE ORDER BY**
Superfund Site,	)	**CONSENT PURSUANT TO**
Cleveland, Ohio	)	**SECTION 106 OF THE**
	)	**COMPREHENSIVE**
	)	**ENVIRONMENTAL RESPONSE,**
Respondents:	)	**COMPENSATION, AND**
	)	**LIABILITY ACT OF 1980,**
Listed in Attachment A	)	**as amended, 42 U.S.C.**
	)	**§9606(a)**
Limited Respondents for	)	
Operation and Maintenance Only)		
Listed in Attachment B	)	

I. JURISDICTION AND GENERAL PROVISIONS

This Order is entered voluntarily by the United States Environmental Protection Agency ("U.S. EPA") and the Respondents. The Order is issued pursuant to the authority vested in the President of the United States by Sections 106(a), 107 and 122 of the Comprehensive Environmental Response, Compensation, and Liability Act of 1980, as amended ("CERCLA"), 42 U.S.C. §§9606(a), 9607 and 9622. This authority has been delegated to the Administrator of the U.S. EPA by Executive Order No. 12580, January 23, 1987, 52 Fed. Reg. 2923, and further delegated to the Regional Administrators by U.S. EPA Delegation Nos. 14-14-A, 14-14-C and 14-14-D, and to the Director, Superfund Division, Region V, by Regional Delegation Nos. 14-14-A, 14-14-C and 14-14-D.

This Order provides for performance of removal actions and reimbursement of response costs incurred by the United States in connection with property located at the former Master Metals, Inc.,facility, 2850 W. Third St., Cleveland, Ohio, (the "MMI Facility")and contamination at and around residential property at 1157, 1159 and 1167 Holmden Avenue, Cleveland, Ohio (the "Holmden Properties"). These areas collectively constitute the "Master Metals Site" or the "Site". This Order requires the Respondents to conduct removal actions described herein to abate an imminent and substantial endangerment to the public health, welfare or the environment that may be presented by the actual or threatened release of hazardous substances at or from the MMI Facility.

A copy of this Order will also be provided to the State of Ohio, which has been notified of the issuance of this Order pursuant to Section 106(a) of CERCLA, 42 U.S.C. §9606(a).

Respondents' participation in this Order will not constitute an admission of liability nor admission of U.S. EPA's findings or determinations contained in this Order except in a proceeding to enforce the terms of this Order. Respondents agree to comply with and be bound by the terms of this Order. Respondents further agree that they will not contest the basis or validity of this Order or its terms.

II. PARTIES BOUND

This Order applies to and is binding upon U.S. EPA, and upon Respondents and Respondents' heirs, receivers, trustees, successors and assigns. Any change in ownership or corporate status of Respondents including, but not limited to, any transfer of assets or real or personal property will not alter such Respondents' responsibilities under this Order. Respondents are jointly and severally liable for carrying out all activities required by this Order except for those activities outlined in this Order that are to be undertaken solely by the Limited Respondents for Operation and Maintenance Only. ~~under Remedy Two.~~ Compliance or noncompliance by one or more Respondents with any provision of this Order will not excuse or justify noncompliance by any other Respondent.

Respondents will ensure that their contractors, subcontractors, and representatives comply with this Order. Respondents will be responsible for any noncompliance with this Order.

III. FINDINGS OF FACT

Based on available information, including the Administrative Record in this matter, U.S. EPA hereby finds that:

1. The Master Metals Site is comprised of both the MMI Facility and a nearby residential property area, the Holmden Properties, where Master Metals lead-bearing materials were deposited as fill.

2. The MMI Facility is located in the "flats" area of downtown Cleveland, in an industrialized sector of the City. This property encompasses 4.3 acres. It is bordered on two

sides by railroad tracks, with an LTV Steel facility located immediately to the east and south. The Cuyahoga River is located approximately 1,500 feet to the east. A playground and athletic field are located approximately 1,500 feet to the west and the nearest residential area begins approximately 2,000 feet to the northwest.

3. The Holmden Properties are located in a residential neighborhood, atop a hillside overlooking the flats. These properties encompass one-half acre. They are surrounded on the north, east and west by continuing residential areas and on the south and southeast by industrial areas located at the bottom of the hillside.

4. Persons, including but not limited to the Respondents listed in Attachment A, arranged for disposal or treatment or arranged with a transporter for transport for disposal or treatment of hazardous substances at the Master Metals Site or accepted hazardous substances for transport to disposal at the Master Metals Site.

5. Persons, including but not limited to the Respondents listed in Attachment A, are current or past owners of the Site, or prior to July 1987 arranged for disposal or treatment, or prior to July 1987 arranged with a transporter for transport for disposal or treatment of hazardous substances at the Site, or accepted hazardous substances for transport to disposal or treatment at the Site or at the Holmden Properties.

6. Respondent NL Industries, Inc. ("NL") initially constructed the MMI Facility in 1932, building it on slag fill. NL owned and operated the MMI Facility as a secondary lead smelter, producing lead alloys from lead-bearing dross and lead scrap materials. NL also engaged in battery cracking as part of its operations.

7. Master Metals purchased the MMI Facility in 1979. Master Metals thereafter continued to run the MMI Facility as a secondary lead smelter, receiving lead-bearing materials from off-Site sources. The lead-bearing feed material received by Master Metals was classified and regulated under the Resource Conservation and Recovery Act ("RCRA"),42 U.S.C. §§ 6901 et seq., as "D008" hazardous waste. In its operations, Master Metals used rotary and pot furnaces to convert these lead-bearing materials into lead ingots. Each furnace used by Master Metals contained a baghouse, a pollution screening structure that collected

particulate matter from the furnace. The collected dust comprised approximately 60 percent lead. The sludge remaining in the furnaces after smelting was classified and regulated under RCRA as "K069" hazardous waste.

8. By-products from the smelting operation included furnace flux, slag, dross, baghouse fines and furnace sludge. Excluding slag, most of the material was recycled back into the furnaces. Slag was tested and disposed of off-site. Cooling water was diverted to the City of Cleveland sewer system. Finished lead ingots were stored in the roundhouse at the north end of the property prior to shipment off-site.

9. Master Metals had a long history of non-compliance with various state and federal environmental, health and safety laws, as well as a history of poor operating practices; releases of hazardous materials to the environment, including the MMI Facility property, have been documented.

10. On November 19, 1980, Master Metals filed a "Part A permit" pursuant to RCRA, thereby obtaining "interim status" under RCRA to operate certain of the MMI Facility's waste piles and treatment units, as well as a container-based storage area.

11. Master Metals filed for Chapter 11 bankruptcy on January 11, 1982, in the United States Bankruptcy Court for the Northern District of Ohio. It subsequently went into reorganization. Prior to November 8, 1985, Master Metals submitted a Part B RCRA application. However, on November 8, 1985, the hazardous waste piles at the MMI Facility that contained lead-bearing dusts lost interim status for failure to comply with financial assurance requirements of 40 C.F.R. Part 265, Subpart H.

12. The United States filed a complaint for violations of RCRA on June 15, 1987, in the United States Bankruptcy Court for the Northern District of Ohio, seeking closure of the D008/K069 waste piles and compliance with RCRA financial responsibility requirements. On September 4, 1987, Master Metals and the United States entered a Stipulation to resolve these RCRA violations.

13. In the late summer of 1987, agents or employees of Master Metals deposited lead-bearing materials from the MMI Facility at the Holmden Properties as fill. These same agents or employees of Master Metals dumped some lead-

bearing materials from the MMI Facility over the edge of the Holmden Properties hillside.

14. In August 1987, Master Metals submitted a partial closure plan to the United States that included procedures to close the D008 and K069 waste piles. Master Metals was to submit an additional closure plan to address all other regulated solid waste management units at a later date. As part of the partial closure plan, Master Metals sampled subsurface soil from the battery storage area waste pile. The soil in this area contained cadmium and lead, but was not considered hazardous according to the U.S. EPA's Environmental Profile ("EP") toxicity criteria. Groundwater between three and ten feet below ground surface contained concentrations of lead.

15. On January 15, 1990, Master Metals entered into a Consent Decree with the United States to resolve continuing RCRA violations. This Consent Decree required, among other things, that Master Metals properly track all hazardous waste at the MMI Facility; submit annual reports to State of Ohio's Environmental Protection Agency("Ohio EPA"); cease battery cracking at the MMI Facility; conduct an investigation to determine subsurface and groundwater conditions at the MMI Facility; characterize waste at the MMI Facility; store the waste properly; close the waste piles containing hazardous waste in accordance with an approved RCRA closure plan; establish closure trust funds or other authorized mechanisms; fund those mechanisms in compliance with RCRA requirements; and establish RCRA required financial liability coverage.

16. Between January 15, 1990, and August 17, 1990, Master Metals accumulated over 1,500 alleged violations of the Consent Decree, spanning 19 decree provisions. Master Metals also committed additional RCRA permit violations during this period, and continued to demonstrate noncompliance with other health and safety standards. These violations included poor handling and control of toxic waste by Master Metals, such that toxic waste remained exposed to the environment at the MMI Facility.

17. In April 1990, Master Metals submitted to the U.S. EPA a revised RCRA "Part B permit" application for closure of various solid waste management units.

18. In August 1990, the United States filed a motion for civil contempt in the District Court for the Northern

District of Ohio regarding Master Metals's Consent Decree violations. The Court denied that motion, granting Master Metals six months to achieve compliance. The United States filed the motion for contempt again in January 1991 with the same result. In May 1991, the Court granted the motion, requiring Master Metals to cease operations in July 1991. However, the Court reconsidered this motion in June and denied the plaintiff government's relief.

19. In addition, on November 9, 1990, the United States demanded by letter $2,286,500 from Master Metals in stipulated penalties for Master Metals Consent Decree violations from January 15, 1990, to August 17, 1990. On June 26, 1992, the United States reached its final determination on these stipulated penalties for Master Metals, reducing Master Metals's stipulated penalty to $1,593,000. Master Metals appealed this determination to the District Court for the Northern District of Ohio pursuant to the Decree's provision on dispute resolution. The District Court, however, never ruled on the penalties. The United States filed a motion to dismiss in October 1996 on the grounds of mootness, which the Court granted in an October 29, 1996 Order.

20. In December 1990, Master Metals contracted with Compliance Technologies, a consulting firm, to install and sample groundwater monitoring wells on the Master Metals Site. Analytical results from the four monitoring wells indicated that the surrounding groundwater was contaminated at levels greater than the maximum contaminant levels (MCLs) for lead and cadmium established under the Safe Drinking Water Act, 42 U.S.C. § 300f et seq.

21. Analysis of MMI Facility soil samples for pH levels and total metals by a U.S. EPA-approved laboratory revealed that the MMI Facility's soil contained elevated levels of barium, cadmium, chromium, lead and nickel. The southern portion of the MMI Facility near the drum storage area contained concentrations of lead exceeding 10,000 parts per million. Elevated lead levels were also discovered near the battery cracking area.

22. In August 1991, Ohio EPA collected samples of raw materials from the Master Metals rotary furnace and two waste bins as part of the Consent Decree requirements. These samples contained lead concentrations as high as 5,349 mg/l.

23. Prior to September 1991, the occupants of 1157 Holmden Avenue at the Holmden Properties contacted Ohio EPA, stating that they believed that Master Metals fill material deposited on their property constituted hazardous waste. The occupants believed that the fill material was hazardous waste because of its distinctive odor and color, because vegetation died and would not grow in the filled area, and because their daughter's feet burned when she walked over the filled area in her bare feet.

24. On September 17, 1991, Ohio EPA began soil sampling at the Holmden Properties. Analysis of these samples by a U.S. EPA approved laboratory showed significant levels of lead and cadmium. Ohio EPA required Master Metals to remove contaminated soils from the Holmden Properties. In March 1992, after the clean-up, Ohio EPA sampled again the soil at the Holmden Properties and discovered additional contamination. Lead was detected in concentrations as high as 7,210 ppm in Holmden Properties soils.

25. In July 1992, U.S. EPA contracted with an outside technical assistance team (TAT) to collect soil samples on and around the MMI Facility property to determine if the MMI Facility contaminants were subject to airborne transport. Analysis of these samples for RCRA metals and Toxicity Characteristic Leachate Procedure (TCLP) metals by a U.S. EPA-approved laboratory revealed that TCLP lead was present in concentrations more than 200 times greater than the RCRA regulatory level of 5 mg/l, at all sample location points except for one MMI Facility location and one location off of the MMI Facility. MMI Facility soil samples indicated the presence of TCLP arsenic and cadmium, with one location testing at 115,000 ppm for lead. Surface samples collected from off of the MMI Facility near both the Valleyview Apartments complex, which is 1,500 feet northwest of the Facility, and near the Tremont Valley Park which is 2,000 feet northwest of the Facility, were found to contain lead concentrations ranging from 148 to 1,850 ppm. The source of this latter lead contamination has not been conclusively traced to the MMI Facility.

26. Three ambient air monitors were installed by the Ohio EPA near the facility property in January of 1992. During the first two quarters of 1992, air samples collected from the station immediately downwind of Master Metals revealed exceedances of the Clean Air Act's National Ambient Air Quality Standards ("NAAQS") for lead, 42 U.S.C. §§ 7401 et seq. In April and May 1992, four more NAAQS violations were

recorded. In July 1992, Master Metals installed a sprinkler system in an attempt to prevent airborne lead from migrating off the MMI Facility property.

27. On August 3, 1992, Ohio EPA ordered an immediate 30-day shut down of the MMI Facility because of Master Metals's "life-threatening" violations of the NAAQS for lead. During Master Metals's shutdown, downwind ambient air monitoring data collected by Ohio EPA registered lead levels in violation of the NAAQS for lead on every day except one. An unknown portion of these NAAQS violations were due to lead-laden MMI Facility dust migrating off of the MMI Facility via prevailing winds. To minimize the effects of wind-blown MMI Facility dust, on September 9, 1992, Master Metals directed a thorough cleaning of the MMI Facility.

28. In December 1992, Master Metals removed additional contaminated soils from the Holmden Properties as ordered by Ohio EPA. After this excavation, Master Metals collected additional soil samples at the Holmden Properties. Analysis of these samples showed elevated levels of lead as high as 57,000 ppm.

29. On August 5, 1993, the Ohio EPA director ordered Master Metals to cease operating the MMI Facility until it could demonstrate compliance. Despite the shutdown of the MMI Facility's furnaces on this date, a U.S. EPA downwind air monitoring station routinely detected elevated lead concentrations as much as 500 times greater than the upwind concentrations and 33 times the NAAQS quarterly average. An unknown portion of these NAAQS violations were due to the lead-laden MMI Facility dust migrating off of the MMI Facility property via prevailing winds.

30. Shortly after Master Metals was shut down, Bank One of Akron, Ohio, took possession of all of Master Metals's cash collateral and accounts receivable.

31. After Master Metals's shutdown, Master Metals and U.S. EPA continued negotiations to resolve Master Metals's RCRA noncompliance. As part of these negotiations, Master Metals and Mr. Mickey, the now-deceased former President of Master Metals, provided financial information to U.S. EPA.

32. On March 28, 1995, U.S. EPA's RCRA Division deferred the Master Metals Site to CERCLA for cleanup. In an August 22, 1995 letter, Master Metals withdrew all permits still in effect regarding its operation, effectively terminating its

ability to legally treat, store or dispose of hazardous waste at the MMI Facility.

33. The occupants of 1157 Holmden Avenue at the Holmden Properties were unable to ever return to their home. The house on the property was vandalized during its vacancy, and later damaged by arson. The City of Cleveland condemned the house on August 18, 1995. On February 22, 1996, the City demolished it.

34. Throughout 1995 and 1996, vandals and scavengers visited the MMI Facility on an intermittent basis. Further, in 1995 or 1996, Master Metals partially demolished one of the MMI Facility structures, leaving piles of rubble, girders and sheet metal standing around the structure's remains.

35. On April 9, 1997, additional Site investigation began at the Holmden Properties. This investigation included sampling which revealed that the Holmden Properties contained approximately 2,000-3,000 cubic yards of lead-impacted materials exceeding the 400 ppm default cleanup criteria set for that investigation. Lead levels as high as 8,350 ppm were detected.

36. Fifty-three potentially responsible parties (the "Smelter Respondents") signed an Administrative Order by Consent for the Master Metals Site, which became effective April 17, 1997, ("Smelter Order"). The Smelter Order required the Smelter Respondents to conduct a time-critical removal action in Phase I. In Phase II the Smelter Order required the Smelter Respondents to complete an Engineering Evaluation and Cost Analysis ("EE/CA")for a non-time-critical removal action for the MMI Facility, pursuant to the National Contingency Plan ("NCP"), 40 C.F.R. Part 300, as amended, and the Superfund Accelerated Cleanup Model ("SACM") guidance. These removal actions were required to abate an imminent and substantial endangerment to the public health, welfare or the environment that may have been presented by the actual or threatened release of hazardous substances at or from the MMI Facility. This order also required the Smelter Respondents in Phase II to prepare an EE/CA report of alternative response actions pursuant to 40 C.F.R. §300.415 (b)(4)(i), and the SACM guidance, to address the remaining environmental concerns at the MMI Facility.

37. On May 13, 1997, the Smelter Respondents submitted a Phase I time-critical removal action workplan for the MMI

Facility to the U.S. EPA for approval. In Phase I, the Smelter Respondents performed the following time-critical removal actions:

a. Analysis and mapping of waste materials and contamination at the MMI Facility for removal purposes;

b. Long-term securing of the MMI Facility against trespassers through the use of fences, signs and other devices, as necessary;

c. Excavation, demolition, consolidation, and/or removal of highly contaminated buildings, structures, soils, loose waste materials, demolition debris, machinery, garbage, dusts, post-industrial debris and office or industrial equipment where such actions reduced the spread of, or direct contact with, the contamination;

d. Removal of drums, barrels, tanks, or other bulk containers that contained or may have contained hazardous substances or pollutants or contaminants where such actions reduced the likelihood of spillage or of exposure to humans, animals or the food chain; and

e. Containment, treatment, disposal, or incineration of hazardous materials, where such action was necessary to reduce the likelihood of human, animal or food chain exposure.

38. On August 8, 1997, the Smelter Respondents submitted the Phase II EE/CA workplan for the MMI Facility to the U.S. EPA for approval. Phase II involved preparing an EE/CA Report identifying and analyzing alternative response actions necessary to complete the non-time critical removal action. The EE/CA was to be consistent with U.S. EPA's guidance entitled, "Guidance on Conducting Non-Time Critical Removal Actions Under CERCLA", EPA/540-R-93-057, Publication 9360.32, dated August 1993.

39. On October 1, 1997, the Smelter Respondents submitted the EE/CA sampling plan for U.S. EPA's approval.

40. On October 23, 1997, six potentially responsible parties ("Holmden Respondents") signed an Administrative Order by Consent for the Holmden Properties("Holmden Order"). The Holmden Order required the Holmden Respondents

to conduct a time-critical removal action at the Holmden Properties pursuant to the NCP and SACM guidance, to abate an imminent and substantial endangerment to the public health, welfare or the environment that may have been presented by the actual or threatened release of hazardous substances at or from the Holmden Properties.

41. On October 15, 1997, the Holmden Respondents submitted a plan of remediation activities for U.S. EPA's approval.

42. On January 19, 1998, the Smelter Respondents submitted the EE/CA data report for the MMI Facility for U.S. EPA's approval.

43. On February 6, 1998, the Holmden Respondents submitted a final report for the removal activities at the Holmden Properties. The Holmden Respondents treated the excavated contaminated soils to below current regulatory levels and below the Land Disposal Restriction level of 0.75 mg/L TCLP for lead. After the removal, the Holmden Respondents restored the Holmden Properties to the properties' original condition including revegetation.

44. On April 24, 1998, the Smelter Respondents submitted the final report for the Phase I time-critical removal activities at the MMI Facility. The Smelter Respondents performed the following actions:

a. Analyzed and mapped all waste materials and contamination for removal purposes, delineating the location of all waste materials and the extent of contaminant toxicity and potential for migration;

b. Secured the MMI Facility against trespassers through the use of fences, signs and other devices, as deemed necessary;

c. Excavated, demolished, consolidated and removed highly contaminated buildings, structures, soils, loose waste materials, loose industrial by-products, construction materials, demolition debris, machinery, garbage, dusts, post-industrial debris and office or industrial equipment;

d. Removed drums, barrels, tanks, and other bulk containers that contained hazardous substances or pollutants or contaminants; and

e. Contained, treated, disposed and incinerated hazardous materials.

Removal activities involved characterizing and removing non-hazardous materials and removing or treating and disposing of hazardous materials. During the course of this project the Smelter Respondents' contractor handled 4,800 cubic yards of solid non-hazardous waste; 500 cubic yards of brick/concrete special waste; 21 tons of asbestos containing material; 1,160 cubic yards of K069, D006, D008 waste; 3,600 cubic yards of chromium trioxide; and over 200 bottles of laboratory chemicals. Over 3,000 gallons of liquid wastes were characterized through the course of this removal.

The result of this time-critical removal action was that all highly contaminated structures were demolished; hazardous materials were characterized and disposed of accordingly; and the MMI Facility was secured to prevent unauthorized entry.

45. On November 23, 1998, the Smelter Respondents submitted the final EE/CA report for the Master Metals Site for U.S. EPA's approval. The Smelter Order Phase II involved completing an EE/CA Report outlining alternative response actions in accordance with the Statement of Work (SOW) attached to the Smelter Order. This SOW required completion of the following tasks:

1. EE/CA Work Plan
2. EE/CA Support Sampling Plan
3. EE/CA Support Sampling
4. EE/CA Data Report
5. EE/CA and Report

46. On November 23, 1998, U.S. EPA reviewed and submitted comments on the revised risk assessment and derivation of the risk based remediation goal for lead documented in the November 23, 1998, Revised EE/CA for the Master Metals Site.

47. On December 10, 1998, U.S. EPA and the Ohio EPA reviewed the revised EE/CA, dated November 23, 1998, for the Master Metals Site. U.S. EPA considered the EE/CA complete and approved it.

48. On February 23, 1999, U.S. EPA submitted a notice of a public comment period on the EE/CA for the clean-up of lead contaminated soils at the MMI Facility, and notice of a March 18, 1999, public meeting on that subject, for

publication in the Cleveland Plain Dealer. U.S. EPA's recommended alternative included:

a. Excavation of off-site contaminated soils;

b. Consolidation of contaminated soils on site;

c. Cover of contaminated areas with two feet of clean fill and revegation;

d. Operation and maintenance of the cover for 30 years; and

e. Deed restrictions to minimize potential exposure to contaminated soil.

49. In March 1999, U.S. EPA released a fact sheet to the citizens of Cleveland and interested stakeholders regarding the EE/CA and U.S. EPA's proposed clean-up plan.

50. On March 18, 1999, U.S. EPA conducted a public meeting regarding the EE/CA and U.S. EPA's proposed clean-up plan. The transcript of the public meeting is in the Administrative Record.

51. On March 31, 1999, U.S. EPA extended the public comment period regarding the EE/CA and U. S. EPA's proposed clean-up plan, for an additional 30 days.

52. In April 1999, U.S. EPA approved the final community involvement plan for the MMI Facility.

53. On May 6, 1999, Ohio EPA approved the City of Cleveland's request for an Urban Setting Designation for the "Industrial Valley Area" within the City of Cleveland. This area includes the Master Metals Site, in the event it is eligible for Ohio EPA's Voluntary Action Program.

54. On August 19, 1999, U. S. EPA identified the community in the area of the MMI Facility as an environmental justice (EJ) area, with the percentage of low income or minority residents greater than or equal to two times the state average. Region 5's EJ criteria percentages for the State of Ohio are a minority population of 13% or greater and a low income population of 60% or greater. In the area near the MMI facility, 26% of the population is minority and 74.2% is low income.

55. On September 30, 1999, U.S. EPA signed an Action Memorandum for a non-time-critical removal action at the MMI Facility.

56. On April 12, 2000, NL surveyed the MMI Facility to facilitate redevelopment by prospective purchasers Bredt-Zanick, LLC and the Northern Ohio Lumber and Timber Company ("NOLTCO") (together the "Prospective Purchasers").

57. On September 22, 2000, U.S. EPA issued a contingent amended Action Memorandum, which changed the project scope from a soil cover cap to an asphalt cap. U.S. EPA did this to accommodate the Prospective Purchasers' planned redevelopment of the MMI Facility. Pursuant to this amended Action Memorandum, if transfer to the Prospective Purchasers does not occur within 60 days of the effective date of this Order as required in Section V, paragraph 2, the change of the project scope will be invalid and the original remedy will be constructed on the Master Metals Site.

58. On May 8, 2001, the Prospective Purchaser Agreement ("PPA") with the Prospective Purchasers became effective. That PPA requires the prospective purchasers to undertake all operation and maintenance for the MMI Facility. ~~if Remedy Two (discussed below) is implemented.~~ Therefore, the prospective purchasers are Limited Respondents for Operation and Maintenance Only. As such, the Prospective Purchasers' only obligation under Section V of this Administrative Order is to perform operation and maintenance. ~~if Remedy Two is implemented.~~ The Limited Respondents for Operation and Maintenance Only shall have no other obligations under this Order, including, but not limited to the obligation to pay costs under Section VII of this Administrative Order.

IV. CONCLUSIONS OF LAW AND DETERMINATIONS

Based on the Findings of Fact set forth above, and the Administrative Record supporting these removal actions, U.S. EPA has determined that:

1. The MMI Facility is a "facility" as defined by Section 101(9) of CERCLA, 42 U.S.C. § 9601(9).

2. Lead, cadmium, chromium, barium and nickel are "hazardous substances" as defined by Section 101(14) of CERCLA, 42 U.S.C. § 9601(14).

3. Each Respondent is a "person" as defined by Section 101(21)of CERCLA, 42 U.S.C. § 9601(21).

4. All Respondents are either persons who at the time of disposal of any hazardous substances owned or operated the MMI Facility, or who arranged for disposal or treatment or transport for disposal or treatment of hazardous substances at the MMI Facility. Each Respondent therefore is liable under Section 107(a) of CERCLA, 42 U.S.C. § 9607(a).

5. The Prospective Purchasers are Limited Respondents for Operation and Maintenance Only, and their only obligations under this Order are to complete the operation and maintenance required by the approved Operation and Maintenance Work Plan discussed in section 2.4 below, Task __ of the SOW, and Section V of the PPA.

6. The conditions described in the Findings of Fact above constitute an actual or threatened "release" of a hazardous substance from the facility into the "environment" as defined by Sections 101(8) and (22) of CERCLA, 42 U.S.C. §§ 9601(8) and (22).

7. The conditions present at the MMI Facility constitute a threat to public health, welfare, or the environment based upon the factors set forth in Section 300.415(b)(2) of the National Oil and Hazardous Substances Pollution Contingency Plan, as amended ("NCP"), 40 C.F.R. § 300.415(b)(2). These factors include, but are not limited to, the following:

   a. Actual or potential exposure to nearby human populations, animals, or the food chain from hazardous substances, pollutants or contaminants; this factor is present at the MMI Facility due to the existence of lead contaminated soils.

   b. High levels of hazardous substances or pollutants or contaminants in soils are largely at or near the surface, that may migrate; this factor is present at the MMI Facility due to the existence of lead contaminated soils.

8. The actual or threatened release of hazardous substances from the MMI Facility may present an imminent and substantial endangerment to the public health, welfare, or the environment within the meaning of Section 106(a) of CERCLA, 42 U.S.C. § 9606(a).

9. The removal actions required by this Order, if properly performed under the terms of this Order, are consistent with the NCP. The removal actions required by this Order are necessary to protect the public health, welfare, or the environment.

V. ORDER

Based upon the foregoing Findings of Fact, Conclusions of Law and Determinations, it is hereby ordered and agreed that Respondents will comply with the following provisions, including but not limited to all documents attached to or incorporated into this Order, and perform the following actions:

1. Designation of Contractor, Project Coordinator, and Remedial Project Manager

Respondents will perform the removal actions required by this Order themselves, or retain one or more contractors to implement the removal actions. Respondents will notify U.S. EPA of Respondents' qualifications or the name and qualifications of such contractor(s), whichever is applicable, within five business days of the effective date of this Order. Respondents will also notify U.S. EPA of the name and qualifications of any other contractors or subcontractors retained to perform work under this Order at least five business days prior to commencement of such work. U.S. EPA retains the right to disapprove of the Respondents or any of the contractors and/or subcontractors retained by the Respondents. If U.S. EPA disapproves a selected contractor, Respondents will retain a different contractor within two business days following U.S. EPA's disapproval and will notify U.S. EPA of that contractor's name and qualifications within three business days of U.S. EPA's disapproval.

Within five business days after the effective date of this Order, the Respondents will designate a Project Coordinator who will be responsible for administration of all the Respondents' actions required by the Order. Respondents will submit the designated coordinator's name, address, telephone number, and qualifications to U.S. EPA. To the greatest extent possible, the Project Coordinator will be present on-site or readily available during site work. U.S. EPA retains the right to disapprove of any Project Coordinator named by the Respondents. If U.S. EPA disapproves a selected Project Coordinator, Respondents will retain a different Project Coordinator within three business days following U.S. EPA's disapproval and will notify U.S. EPA of that person's name and qualifications within four business days of U.S. EPA's disapproval. Receipt by Respondents' Project

Coordinator of any notice or communication from U.S. EPA relating to this Order will constitute receipt by all Respondents.

The U.S. EPA has designated Gwendolyn Massenburg of the Remedial Response Branch, Region V, as its Remedial Project Manager ("RPM"). Respondents will direct all submissions required by this Order to the RPM at 77 West Jackson Boulevard, SR-6J, Chicago, Illinois, 60604-3590, by certified or express mail. Respondents will also send a copy of all submissions to Susan Prout, Associate Regional Counsel, 77 West Jackson Boulevard, C-14J, Chicago, Illinois, 60604-3590,and to the Ohio EPA, attention: Sheila Abraham, Division of Emergency and Remedial Response, 2110 East Aurora Road, Twinsburg, OH 44087. All Respondents are encouraged to make their submissions to U.S. EPA on recycled paper (which includes significant post consumer waste paper content where possible) and using two-sided copies.

U.S. EPA and Respondents will have the right, subject to the immediately preceding paragraph, to change their designated Project Coordinator, RPM or Project Counsel. U.S. EPA will notify the Respondents, and Respondents will notify U.S. EPA, as early as possible before such a change is made, but in no case less than twenty four hours before such a change. The initial notification may be made orally but it will be promptly followed by a written notice.

2. Work to Be Performed

If Prospective Purchasers acquire ownership of the MMI Facility within 60 calendar days of the effective date of this Order, Respondents will perform the actions set forth in ~~"Remedy Two"~~ below. ~~If Prospective Purchasers do not acquire the ownership of the MMI Facility within 60 calendar days of the effective date of this Order, Respondents will perform the actions set forth in "Remedy One," below.~~

~~**Remedy One:**~~ Respondents will perform, at a minimum, the following removal actions:

1. Remove site fencing.

2. Excavate perimeter soil (eastern, western, and southern boundary) to a depth to obtain a total lead concentration of 1000mg/kg (ppm) or to the depth where the original slag is reached to reduce the likelihood of human, animal or food chain exposure.

3. Conduct a treatability study of all material excavated to determine if treatment of this material is a viable option. Treatment of this material is required when the excavated soil does not pass TCLP. Respondents will provide a copy of the treatability study to U. S. EPA prior to consolidation of the soils. See Section 1.1 of the Statement of Work for treatment of the excavated soils.

4. Perform treatment (if necessary) in secondary containers or cans using the lead stabilization process. Treatment will satisfy the Land Disposal Restriction prior to consolidation. See section 1.1 of the Statement of Work for treatment requirements. Respondents will submit a post-treatment report to U.S. EPA prior to consolidating the material on site.

5. Backfill all areas excavated or sub-graded areas to grade with clean soil. The existing property lines will serve as center and highest elevation point of the graded slope.

6. Consolidate excavated treated soils and Holmden Properties treated soils on-site, underneath an impermeable geomembrane, or appropriately dispose of the material in a hazardous waste landfill or in a solid waste landfill.

~~7. Cover the impermeable geomembrane with two feet of revegetated top soil. Only the most severely deteriorated portions of the property will be placed under the cover system.~~

7. The site must be capped with the asphalt cover system, engineered (with the necessary thickness and load-bearing capacity) to permit appropriate reuse, as specified int the SOW. ~~Cover the impermeable geomembrane for grading purposes only with clean fill material and cover the clean fill material with asphalt to support redevelopment or reuse. Only the most severely deteriorated portions of the property will be placed under the cover system.~~

8. Provide specifics on the cover system and on the areas under the cover system (including a cross section and designation of the areas where the treated soils will be placed) in the remedial design plan for U.S. EPA and Ohio EPA approval.

9. Repair or recondition the cracked concrete (defined as fully penetrating the existing concrete surfaces with a width greater than ½ inch) portions of the MMI Facility by sealing the cracks followed by scarification or encapsulation of the concrete surface.

10. Eliminate dangers associated with open pits and sumps on the MMI Facility.

11. Replace the fence on the MMI Facility as specified in the SOW. ~~with industrial grade fence topped with three strands of barbed wire.~~

12. Perform required operation and maintenance as required for the next thirty years. The particular obligations of the Respondents and the Limited Respondent for Operation and Maintenance Only are set forth in Section V.2.4 below.

~~**Remedy Two:** Respondents will perform, at a minimum, the following removal actions:~~

~~1. All removal actions in Remedy One, above, except as noted below.~~

~~2. Replace Paragraph 7, above, with the following Paragraph 7:~~

~~7. Cover the impermeable geomembrane for grading purposes only with clean fill material and cover the clean fill material with asphalt to support redevelopment or reuse. Only the most severely deteriorated portions of the property will be placed under the cover system.~~

2.1 Work Plan and Implementation

Attached to this Order for the Respondents to follow is a Statement of Work.

Within sixty business days after the effective date of this Order, the Respondents will submit to U.S. EPA for approval, a draft Work Plan for performing the removal activities set forth above. The draft Work Plan will provide a description of, and an expeditious schedule for, the actions required by this Order.

U.S. EPA may approve, disapprove, require revisions to, or modify the draft Work Plan. If U.S. EPA requires revisions,

Respondents will submit a revised draft Work Plan within seven business days of receipt of U.S. EPA's notification of required revisions. Respondents will implement the Work Plan as finally approved in writing by U.S. EPA in accordance with the schedule approved by U.S. EPA. Once approved, or approved with modifications, the Work Plan, the schedule, and any subsequent modifications will be fully enforceable under this Order. Respondents will notify U.S. EPA at least forty eight hours prior to performing any on-site work pursuant to the U.S. EPA approved Work Plan. Respondents will not commence or undertake any removal actions at the Site without prior U.S. EPA approval.

2.2 Health and Safety Plan

Within thirty business days after the effective date of this Order, the Respondents will submit for U.S. EPA review and comment a plan that ensures the protection of the public health and safety during the performance of on-site work under this Order. This plan will comply with applicable Occupational Safety and Health Administration ("OSHA") regulations found at 29 C.F.R. Part 1910. If U.S. EPA determines it is appropriate, the plan will also include contingency planning. Respondents will incorporate all changes to the plan recommended by U.S. EPA, and implement the plan during the pendency of the removal action.

2.3 Quality Assurance and Sampling

All sampling and analysis performed pursuant to this Order will conform to U.S. EPA direction, approval, and guidance regarding sampling, quality assurance/quality control ("QA/QC"), data validation, and chain of custody procedures. Respondents will ensure that the laboratory used to perform the analysis participates in a QA/QC program that complies with U.S. EPA guidance.

Upon request by U.S. EPA, Respondents will have such a laboratory analyze samples submitted by U.S. EPA for quality assurance monitoring. Respondents will provide to U.S. EPA the quality assurance/quality control procedures followed by all sampling teams and laboratories performing data collection and/or analysis. Respondents will also ensure provision of analytical tracking information consistent with OSWER Directive No. 9240.0-2B, "Extending the Tracking of Analytical Services to PRP-Lead Superfund Sites."

Upon request by U.S. EPA, Respondents will allow U.S. EPA or its authorized representatives to take split and/or duplicate samples of any samples collected by Respondents or their

contractors or agents while performing work under this Order. Respondents will notify U.S. EPA not less than three business days in advance of any sample collection activity. U.S. EPA will have the right to take any additional samples that it deems necessary.

2.4 Post-Removal Site Control/Operation and Maintenance

a. In accordance with the Work Plan schedule, or as otherwise directed by the RPM, Respondents will submit a proposal for post-removal site control, consistent with Section 300.415(l) of the NCP, 40 C.F.R. §300.415(l), and OSWER Directive 9360.2-02. The Limited Respondents for Operation and Maintenance Only, are primarily responsible for completing the post-removal site control and Operation and Maintenance of the MMI Facility. ~~if Remedy Two is implemented. The Respondents are primarily responsible for post-removal site control and operation and maintenance of the MMI Facility if Remedy One is implemented. In addition,~~ The Respondents are secondarily responsible for operation and maintenance, ~~if Remedy Two is implemented,~~ except that they are not responsible for maintaining the ~~asphalt cap~~cover system under any circumstances.

b. The Respondents will also make a payment of $9600 to satisfy their obligation to perform Operation and Maintenance of the cover system~~soil cap. under Remedy One if Remedy Two is implemented.~~

c. Respondents and Limited Respondents for Operation and Maintenance Only will provide U.S. EPA with documentation of all post-removal site control arrangements.

2.5 Reporting

Respondents will submit a monthly written progress report to U.S. EPA concerning actions undertaken pursuant to this Order, beginning the 10th day of each month following the date of U.S. EPA's approval of the Work Plan, until termination of this Order, unless otherwise directed in writing by the RPM. These reports will describe all significant developments during the preceding period, including the work performed and any problems encountered, analytical data received during the reporting period, and developments anticipated during the next reporting period, including a schedule of work to be performed, anticipated

problems, and planned resolutions of past or anticipated problems.

Any Respondent that owns any portion of the Site will, at least thirty days prior to the conveyance of any interest in real property at the Site, give written notice of this Order to the transferee and written notice of the proposed conveyance to U.S. EPA and the State. The notice to U.S. EPA and the State will include the name and address of the transferee. The party conveying such an interest will require that the transferee will provide access as described in Section V.3 (Access to Property and Information).

2.6 Final Report

Within sixty calendar days after completion of all removal actions required under this Order, the Respondents will submit for U.S. EPA review a final report summarizing the actions taken to comply with this Order. The final report will conform to the requirements set forth in Section 300.165 of the NCP, 40 C.F.R. §300.165. The final report will also include a good faith estimate of total costs incurred in complying with the Order, a listing of quantities and types of materials removed off-site or handled on-site, a discussion of removal and disposal options considered for those materials, a listing of the ultimate destinations of those materials, a presentation of the analytical results of all sampling and analyses performed, and accompanying appendices containing all relevant documentation generated during the removal action (e.g., manifests, invoices, bills, contracts, and permits).

The final report will also include the following certification signed by a person who supervised or directed the preparation of that report:

> Under penalty of law, I certify that, to the best of my knowledge, after appropriate inquiries of all relevant persons involved in the preparation of this report, the information submitted is true and complete.

3. Access to Property and Information

~~Respondents, if Remedy One is implemented, and~~ Limited Respondents for Operation and Maintenance Only~~, if Remedy Two is implemented,~~ will use best efforts to provide or obtain access to the MMI Facility and off-site areas to which access is necessary to implement this Order, and will provide access to all records and documentation related to the conditions at the MMI Facility

and the actions conducted pursuant to this Order. Such access will be provided to U.S. EPA employees, contractors, agents, consultants, designees, representatives, and State of Ohio representatives. These individuals will be permitted to move freely at the MMI Facility and appropriate off-site areas in order to conduct actions which U.S. EPA determines to be necessary. Respondents will submit to U.S. EPA, upon request, the results of all sampling or tests and all other validated data generated by Respondents or their contractors, or on the Respondents' behalf during implementation of this Order.

Where work under this Order is to be performed in areas owned by or in possession of someone other than Respondents, ~~Respondents if Remedy One is implemented, and~~ Limited Respondents for Operation and Maintenance Only~~, if Remedy Two is implemented,~~ will use their best efforts to obtain all necessary access agreements within thirty calendar days after the effective date of this Order, or as otherwise specified in writing by the RPM. Respondents will immediately notify U.S. EPA if, after using their best efforts, they are unable to obtain such agreements. Respondents will describe in writing their efforts to obtain access. Upon Respondents' written request, U.S. EPA may then assist Respondents in gaining access, to the extent necessary to effectuate the response actions described herein, using such means as U.S. EPA deems appropriate. Respondents will reimburse the United States for all costs and attorneys' fees incurred by the United States in obtaining such access.

4. Record Retention, Documentation, Availability of Information

Respondents will preserve all documents and information, in their possession or the possession of their contractors, subcontractors or representatives, relating to work performed under this Order, or relating to the hazardous substances found on or released from the MMI Facility, for six years following completion of the removal actions required by this Order. At the end of this six year period and at least sixty days before any document or information is destroyed, Respondents will notify U.S. EPA that such documents and information are available to U.S. EPA for inspection, and upon request, will provide the originals or copies of such documents and information to U.S. EPA. In addition, Respondents will provide documents and information retained under this Section at any time before expiration of the six year period at the written request of U.S. EPA. Any information that Respondents are required to provide or maintain pursuant to this Order is not subject to the Paperwork Reduction Act of 1995, 44 U.S.C. §3501 et seq.

5. Off-Site Shipments

All hazardous substances, pollutants or contaminants removed off-site pursuant to this Order for treatment, storage or disposal will be treated, stored, or disposed of at a facility in compliance, as determined by U.S. EPA, with the U.S. EPA Off-Site Rule, 40 C.F.R. §300.440, 58 Fed. Reg. 49215 (Sept. 22, 1993).

6. Compliance With Other Laws

Respondents will perform all actions required pursuant to this Order in accordance with all applicable local, state, and federal laws and regulations except as provided in Section 121(e) of CERCLA, 42 U.S.C. §9621(e), and 40 C.F.R. §300.415(j). In accordance with 40 C.F.R. §300.415(j), all on-site actions required pursuant to this Order will, to the extent practicable, as determined by U.S. EPA, considering the exigencies of the situation, attain applicable or relevant and appropriate requirements under federal environmental or state environmental or facility siting laws.

7. Emergency Response and Notification of Releases

If any incident, or change in Site conditions, during the activities conducted pursuant to this Order causes or threatens to cause an additional release of hazardous substances from the MMI Facility or an endangerment to the public health, welfare, or the environment, the Respondents will immediately take all appropriate action to prevent, abate or minimize such release or endangerment caused or threatened by the release. Respondents will also immediately notify the RPM or, in the event of his/her unavailability, will notify the Regional Duty Officer, Emergency Response Branch, Region V at (312) 353-2318, of the incident or Site conditions. If Respondents fail to respond, U.S. EPA may respond to the release or endangerment and reserve the right to recover costs associated with that response.

Respondents will submit a written report to U.S. EPA within seven business days after each release, setting forth the events that occurred and the measures taken or to be taken to mitigate any release or endangerment caused or threatened by the release and to prevent the reoccurrence of such a release. Respondents will also comply with any other notification requirements, including those in Section 103 of CERCLA, 42 U.S.C. §9603, and Section 304 of the Emergency Planning and Community Right-To-Know Act, 42 U.S.C. §11004.

8. Institutional Controls

If the MMI Facility, or any other property where access and/or land/water use restrictions are needed to implement this Order, is owned or controlled by persons other than any of the ~~Respondents, Respondents if Remedy One is implemented and~~ Limited Respondents for Operation and Maintenance Only ~~if Remedy Two is implemented~~ shall use best efforts to secure from such persons:

a. an agreement, enforceable by Respondents, Limited Respondents for Operation and Maintenance Only, and U.S. EPA, to refrain from using the MMI Facility, or such other property, in any manner that would interfere with or adversely affect the integrity or protectiveness of the actions to be implemented pursuant to this Order. Such restrictions include, but are not limited to,

1. Well construction: no person may construct or reconstruct a well on the property without:

   (a) notifying U.S. EPA and Ohio EPA;

   (b) determining what specific prohibitions or requirements are applicable to the well;

   (c) obtaining approval from all relevant authorities and U.S. EPA prior to the construction or reconstruction; and

   (d) complying with all requirements applicable to the well.

2. Drilling into the cover system~~asphalt or soil cover~~: no person may drill or puncture the ~~asphalt or soil~~ cover system on the property without:

   (a) notifying U.S. EPA and Ohio EPA;

   (b) determining what specific prohibitions or requirements are applicable to the asphalt or soil cover;

   (c) obtaining approval from all relevant authorities and U.S. EPA prior to the drilling; and

   (d) maintaining the protectiveness of the asphalt or soil cover.

3. Restricted activities: no person may undertake the

following activities without written permission from U.S. EPA:

(a) excavating or grading of any portion of the land surface within the current fence line; ~~including in the area located at the fence line located at the rear of the site and any area where the Phase 1 excavation occurred;~~

(b) filling in the capped area;

(c) constructing or installing a building or other structures with a foundation that would sit on or be placed within the cap or cover; or

(d) using of groundwater for drinking purposes.

b. The execution and recordation in the Recorder's Office of Cuyahoga County, State of Ohio, of an easement, running with the land, that (i) grants a right of access as set forth at Section V.3 of this Order, above; and (ii) grants the right to enforce the land/water use restrictions listed in Section V.8 of this Order, or other restrictions that U.S. EPA determines are necessary to implement, ensure non-interference with, or ensure the protectiveness of the actions to be performed pursuant to this Order. The access rights and/or rights to enforce land/water use restrictions shall be granted to one or more of the following persons, as determined by U.S. EPA: (i) the United States, on behalf of EPA, and its representatives, (ii) the State and its representatives, (iii) the Respondents and their representatives, and/or (iv) other appropriate grantees. Within forty-five days of entry of this Order, ~~the Respondents (if Remedy One) or~~ the Limited Respondents for Operation and Maintenance Only ~~(if Remedy Two)~~ shall submit to U.S. EPA for review and approval with respect to such property:

1. A draft easement enforceable under the laws of the State of Ohio, free and clear of all prior liens and encumbrances (except as approved by U.S. EPA), and acceptable under the Attorney General's Title Regulations promulgated pursuant to 40 U.S.C. § 255; and

2. A current title commitment or report prepared in accordance with the U.S. Department of Justice Standards for the Preparation of Title Evidence in Land Acquisitions by the United States (1970) (the "Standards").

Within fifteen days of EPA's approval and acceptance of the

easement, ~~Respondents (if Remedy One) or~~ Limited Respondents for Operation and Maintenance Only ~~(if Remedy Two)~~ shall update the title search and, if it is determined that nothing has occurred since the effective date of the commitment or report to affect the title adversely, the easement shall be recorded with the Recorder's Office of Cuyahoga County. Within thirty days of the recording of the easement, the ~~Respondents (if Remedy One) or~~ Limited Respondents for Operation and Maintenance Only ~~(if Remedy Two)~~ shall provide EPA with final title evidence acceptable under the Standards, and a certified copy of the original recorded easement showing the clerk's recording stamps.

~~Respondents (if Remedy One) or~~ Limited Respondents for Operation and Maintenance Only ~~(if Remedy Two)~~ will immediately notify U.S. EPA if, after using their best efforts, they are unable to obtain such agreements. Respondents will describe in writing their efforts to obtain access. Upon Respondents' written request U.S. EPA may then assist ~~Respondents (if Remedy One) or~~ Limited Respondents for Operation and Maintenance Only ~~(if Remedy Two)~~ in gaining access, to the extent necessary to effectuate the response actions described herein, using such means as U.S. EPA deems appropriate. Respondents will reimburse U.S. EPA for all costs and attorneys' fees incurred by the United States in obtaining such access.

If U.S. EPA determines that land/water use restrictions in the form of state or local laws, regulations, ordinances or other governmental controls are needed to implement this Order's actions, ensure the integrity and protectiveness thereof, or ensure non-interference therewith, ~~Respondents (if Remedy One) or~~ Limited Respondents for Operation and Maintenance Only ~~(if Remedy Two)~~ shall cooperate with U.S. EPA's efforts to secure such governmental controls.

VI. AUTHORITY OF THE U.S. EPA REMEDIAL PROJECT MANAGER

The Remedial Project Manager (RPM) will be responsible for overseeing the implementation of this Order. The RPM will have the authority vested in an RPM by the NCP, including the authority to halt, conduct, or direct any work required by this Order, or to direct any other response action undertaken by U.S. EPA or Respondents at the Site. Absence of the RPM from the MMI Facility will not be cause for stoppage of work unless specifically directed by the RPM.

VII. REIMBURSEMENT OF COSTS

~~If Remedy Two is implemented,~~ Respondents will pay $62,760 in settlement of all costs that have accrued through January 31, 2001. ~~If Remedy One is implemented, Respondents will pay~~

~~$125,519.09 for costs incurred through January 31, 2001.~~

In addition, U.S. EPA will send Respondents a bill for "oversight costs" on an annual basis, such bill to include an Itemized Cost Summary. "Oversight costs" are all costs, including, but not limited to, direct and indirect costs, that the United States incurs in reviewing or developing plans, reports and other items pursuant to this AOC. "Oversight costs" will also include all costs, including direct and indirect costs, incurred by the United States in connection with the Site starting from February 1, 2001.

Respondents will, within thirty calendar days of receipt of a bill, remit a cashier's or certified check for the amount of the bill made payable to the "Hazardous Substance Superfund," to the following address:

U.S. Environmental Protection Agency
Program Accounting & Analysis Section
P.O. Box 70753
Chicago, Illinois 60673

Respondents will simultaneously transmit a copy of the check to the Director, Superfund Division, U.S. EPA Region 5, 77 West Jackson Blvd., Chicago, Illinois, 60604-3590. Payments will be designated as "Response Costs - Master Metals Cleveland Site" and will reference:

the payer's name and address;

the U.S. EPA site identification number 05WB; and

the docket number of this Order.

In the event that any payment is not made within the deadlines described above, Respondents will pay interest on the unpaid balance. Interest is established at the rate specified in Section 107(a) of CERCLA, 42 U.S.C. §9607(a). The interest will begin to accrue on the date of the Respondents' receipt of the bill (or for the $$62,760 due under this Order, on the effective date of this Order). Interest will accrue at the rate specified through the date of the payment. Payments of interest made under this paragraph will be in addition to such other remedies or sanctions available to the United States by virtue of Respondents' failure to make timely payments under this Section.

Respondents may dispute all or part of a bill for Oversight costs submitted under this Order, if Respondents allege that U.S. EPA has made an accounting error, or if Respondents allege that a cost item is inconsistent with the NCP.

If any dispute over costs is resolved before payment is due, the amount due will be adjusted as necessary. If the dispute is

not resolved before payment is due, Respondents will pay the full amount of the uncontested costs into the Hazardous Substance Fund as specified above on or before the due date. Within the same time period, Respondents will pay the full amount of the contested costs into an interest-bearing escrow account. Respondents will simultaneously transmit a copy of both checks to the RPM. Respondents will ensure that the prevailing party or parties in the dispute will receive the amount upon which they prevailed from the escrow funds plus interest within twenty calendar days after the dispute is resolved.

VIII. DISPUTE RESOLUTION

The parties to this Order will attempt to resolve, expeditiously and informally, any disagreements concerning this Order.

If the Respondents object to any U.S. EPA action taken pursuant to this Order, including billings for oversight costs, the Respondents will notify U.S. EPA in writing of their objections within ten calendar days of such action, unless the objections have been informally resolved. This written notice will include a statement of the issues in dispute, the relevant facts upon which the dispute is based, all factual data, analysis or opinion supporting Respondents' position, and all supporting documentation on which such party relies. U.S. EPA will submit its Statement of Position, including supporting documentation, no later than ten calendar days after receipt of the written notice of dispute. In the event that these ten-day time periods for exchange of written documents may cause a delay in the work, they will be shortened upon, and in accordance with, notice by U.S. EPA. The time periods for exchange of written documents relating to disputes over billings for oversight costs may be extended at the sole discretion of U.S. EPA.

An administrative record of any dispute under this Section will be maintained by U.S. EPA. The record will include the written notification of such dispute, and the Statement of Position served pursuant to the preceding paragraph. Upon review of the administrative record, the Director of the Superfund Division, U.S. EPA Region V, will resolve the dispute consistent with the NCP and the terms of this Order.

Respondents' obligations under this Order will not be tolled by submission of any objection for dispute resolution under this Section. Following resolution of the dispute, as provided by this Section, Respondents will fulfill the requirement that was the subject of the dispute in accordance with the agreement reached or with U.S. EPA's decision, whichever occurs.

IX. FORCE MAJEURE

Respondents agree to perform all requirements under this Order within the time limits established under this Order, unless the performance is delayed by a force majeure. For purposes of this Order, a force majeure is defined as any event arising from causes beyond the control of Respondents or of any entity controlled by Respondents, including but not limited to their contractors and subcontractors, that delays or prevents performance of any obligation under this Order despite Respondents' best efforts to fulfill the obligation. Force majeure does not include financial inability to complete the work or increased cost of performance.

Respondents will notify U.S. EPA orally within twenty-four hours after Respondents become aware of any event that Respondents contend constitutes a force majeure, and in writing within seven calendar days after the event. Such notice will: identify the event causing the delay or anticipated delay; estimate the anticipated length of delay, including necessary demobilization and re-mobilization; state the measures taken or to be taken to minimize the delay; and estimate the timetable for implementation of the measures. Respondents will take all reasonable measures to avoid and minimize the delay. Failure to comply with the notice provision of this Section will be grounds for U.S. EPA to deny Respondents an extension of time for performance. Respondents will have the burden of demonstrating by a preponderance of the evidence that the event is a force majeure, that the delay is warranted under the circumstances, and that best efforts were exercised to avoid and mitigate the effects of the delay.

If U.S. EPA determines a delay in performance of a requirement under this Order is or was attributable to a force majeure, the time period for performance of that requirement will be extended as deemed necessary by U.S. EPA. Such an extension will not alter Respondents' obligation to perform or complete other tasks required by the Order which are not directly affected by the force majeure.

X. STIPULATED AND STATUTORY PENALTIES

For each day, or portion thereof, that Respondents fail to fully perform any requirement of this Order in accordance with the schedule established pursuant to this Order, Respondents will be liable as follows:

Deliverable/Activity	Penalty For Days 1-7	Penalty For More Than 7 Days

Failure to Submit a Draft or Revised Work Plan	$750/Day	$2,000/Day
Late Submittal of Progress Reports or Other Miscellaneous Reports/Submittals	$200/Day	$500/Day
Failure to Meet any Scheduled Deadline in the Order	$200/Day	$500/Day
Failure to Meet of the Operation and Maintenance Requirements, if applicable	$200/Day	$500/Day

Upon receipt of written demand by U.S. EPA, Respondents will make payment to U.S. EPA within twenty days and interest will accrue on late payments in accordance with Section VII of this Order (Reimbursement of Costs).

Even if violations are simultaneous, separate penalties will accrue for separate violations of this Order. Penalties accrue and are assessed per violation per day. Penalties will accrue regardless of whether EPA has notified Respondents of a violation or act of noncompliance. The payment of penalties will not alter in any way Respondents' obligations to complete the performance of the work required under this Order. Stipulated penalties will accrue, but need not be paid, during any dispute resolution period concerning the particular penalties at issue. If Respondents prevail upon resolution, Respondents will pay only such penalties as the resolution requires. In its unreviewable discretion, U.S. EPA may waive its rights to demand all or a portion of the stipulated penalties due under this Section. Such a waiver must be made in writing.

Violation of any provision of this Order may subject Respondents to civil penalties of up to $27,500 per violation per day, as provided in Section 106(b)(1) of CERCLA, 42 U.S.C. §9606(b)(1). Respondents may also be subject to punitive damages in an amount up to three times the amount of any cost incurred by the United States as a result of such violation, as provided in Section 107(c)(3) of CERCLA, 42 U.S.C. §9607(c)(3). Should Respondents violate this Order or any portion hereof, U.S. EPA may carry out the required actions unilaterally, pursuant to Section 104 of CERCLA, 42 U.S.C. §9604, and/or may seek judicial enforcement of this Order pursuant to Section 106 of CERCLA, 42 U.S.C. §9606.

XI. RESERVATION OF RIGHTS

Except as specifically provided in this Order, nothing herein will limit the power and authority of U.S. EPA or the United States to take, direct, or order all actions necessary to protect public health, welfare, or the environment or to prevent, abate, or minimize an actual or threatened release of hazardous substances, pollutants or contaminants, or hazardous or solid waste on, at, or from the Site. Further, nothing herein will prevent U.S. EPA from seeking legal or equitable relief to enforce the terms of this Order. U.S. EPA also reserves the right to take any other legal or equitable action as it deems appropriate and necessary, or to require the Respondents in the future to perform additional activities pursuant to CERCLA or any other applicable law. Except as specifically provided in this Order, Respondents reserve the right to assert any factual or legal position in any action taken by U.S. EPA or the United States under this Article XI.

XII. OTHER CLAIMS

By issuance of this Order, the United States and U.S. EPA assume no liability for injuries or damages to persons or property resulting from any acts or omissions of Respondents. The United States or U.S. EPA will not be a party or be held out as a party to any contract entered into by the Respondents or their directors, officers, employees, agents, successors, representatives, assigns, contractors, or consultants in carrying out activities pursuant to this Order. Each party will bear its own costs and attorneys fees in connection with the action resolved by this Order.

Except as expressly provided in Section XIII (Covenant Not To Sue), nothing in this Order constitutes a satisfaction of or release from any claim or cause of action against the Respondents or any person not a party to this Order, for any liability such person may have under CERCLA, other statutes, or the common law, including but not limited to any claims of the United States for costs, damages and interest under Sections 106(a) or 107(a) of CERCLA, 42 U.S.C. §§9606(a), 9607(a).

This Order does not constitute a preauthorization of funds under Section 111(a)(2) of CERCLA, 42 U.S.C. §9611(a)(2). The Respondents waive any claim to payment under Sections 106(b), 111, and 112 of CERCLA, 42 U.S.C. §§9606(b), 9611, and 9612, against the United States or the Hazardous Substance Superfund arising out of any action performed under this Order.
No action or decision by U.S. EPA pursuant to this Order will give rise to any right to judicial review except as set forth in

Section 113(h) of CERCLA, 42 U.S.C. §9613(h).

XIII. COVENANT NOT TO SUE

Except as otherwise specifically provided in this Order, upon issuance of the U.S. EPA notice referred to in Section XVII (Notice of Completion), U.S. EPA covenants not to sue Respondents for judicial imposition of damages or civil penalties or to take administrative action against Respondents for any failure to perform removal actions agreed to in this Order except as otherwise reserved herein.

Except as otherwise specifically provided in this Order, in consideration and upon Respondents' payment of the response costs specified in Section VII of this Order, U.S. EPA covenants not to sue or to take administrative action against Respondents under Section 107(a) of CERCLA, 42 U.S.C. §9607(a), for recovery of past and oversight costs incurred by the United States in connection with this removal action and this Order. This covenant not to sue will take effect upon the receipt by U.S. EPA of the payments required by Section VII (Reimbursement of Costs).

These covenants not to sue are conditioned upon the complete and satisfactory performance by Respondents of their obligations under this Order. These covenants not to sue extend only to the Respondents and do not extend to any other person.

XIV. CONTRIBUTION PROTECTION

With regard to claims for contribution against Respondents for matters addressed in this Order, the Parties hereto agree that the Respondents are entitled to protection from contribution actions or claims to the extent provided by Section 113(f)(2) and 122(h)(4) of CERCLA, 42 U.S.C. §§9613(f)(2) and 9622(h)(4).

Nothing in this Order precludes Parties from asserting any claims, causes of action or demands against any persons not parties to this Order for indemnification, contribution, or cost recovery.

XV. INDEMNIFICATION

Respondents agree to indemnify, save and hold harmless the United States, its officials, agents, contractors, subcontractors, employees and representatives from any and all claims or causes of action: (A) arising from, or on account of, acts or omissions of Respondents and Respondents' officers, heirs, directors, employees, agents, contractors, subcontractors, receivers, trustees, successors or assigns, in carrying out actions pursuant to this Order; and (B) for damages or reimbursement arising from or on account of any contract,

agreement, or arrangement between (any one or more of) Respondents, and any persons for performance of work on or relating to the Site, including claims on account of construction delays. Nothing in this Order, however, requires indemnification by Respondents for any claim or cause of action against the United States based on negligent action taken solely and directly by U.S. EPA (not including oversight or approval of plans or activities of the Respondents).

XVI. MODIFICATIONS

Modifications to any plan or schedule may be made in writing by the RPM or at the RPM's oral direction. If the RPM makes an oral modification, it will be memorialized in writing within 7 business days; however, the effective date of the modification will be the date of the RPM's oral direction. Any other requirements of this Order may be modified in writing by mutual agreement of the parties.

If Respondents seek permission to deviate from any approved plan or schedule, Respondents' Project Coordinator will submit a written request to U.S. EPA for approval outlining the proposed modification and its basis.

No informal advice, guidance, suggestion, or comment by U.S. EPA regarding reports, plans, specifications, schedules, or any other writing submitted by the Respondents will relieve Respondents of their obligations to obtain such formal approval as may be required by this Order, and to comply with all requirements of this Order unless it is formally modified.

XVII. NOTICE OF COMPLETION

When U.S. EPA determines, after U.S. EPA's review of the Final Report, that all work has been fully performed in accordance with this Order, except for certain continuing obligations required by this Order (e.g., record retention, payment of costs), U.S. EPA will provide written notice to the Respondents. If U.S. EPA determines that any removal activities have not been completed in accordance with this Order, U.S. EPA will notify the Respondents, provide a list of the deficiencies, and require that Respondents modify the Work Plan if appropriate to correct such deficiencies. The Respondents will implement the modified and approved Work Plan and will submit a modified Final Report in accordance with the U.S. EPA notice. Failure to implement the approved modified Work Plan will be a violation of this Order.

XVIII. SEVERABILITY

If a court issues an order that invalidates any provision of

this Order or finds that Respondents have sufficient cause not to comply with one or more provisions of this Order, Respondents will remain bound to comply with all provisions of this Order not invalidated by the court's order.

XIX. EFFECTIVE DATE

This Order will be effective upon receipt by NL of a copy of this Order signed by the Director, Superfund Division, U.S. EPA Region V.

IN THE MATTER OF:

Master Metals, Inc.,
Superfund Site,
Cleveland, Ohio

SIGNATORIES

Each undersigned representative of a signatory to this Administrative Order on Consent certifies that he or she is fully authorized to enter into the terms and conditions of this Order and to bind such signatory, its successors and assigns, to this document.

Agreed this _____ day of _____, _____.

By: _____
(Signature)

Name: _____

Position: _____

Signatory: _____

IN THE MATTER OF:

Master Metals, Inc.,
Superfund Site,
Cleveland, Ohio

IT IS SO ORDERED AND AGREED

BY:_____ DATE:_____

William E. Muno, Director
Superfund Division
United States
Environmental Protection Agency
Region 5

APPENDIX B

FINAL PERFORMANCE STANDARD VERIFICATION PLAN

1.0 INTRODUCTION

The Performance Standard Verification Plan (PSVP) is designed to ensure that both short-term and long-term performance standards are met for the removal design and removal action (RD/RA) at the Master Metals Superfund Site (MMI) site in Cleveland, Ohio. This PSVP, a part of the RD/RA Work Plan, references the Contingency Plan, the Field Sampling Plan, the Health and Safety Plan and the Quality Assurance Project Plan. The PSVP describes the methods that will be used to sample and analyze in-place soils, treated soils, air, and backfill.

1.1 Coordinating Documents

The plans that will be prepared and submitted during the course of this project include the following:

- RD/RA Work Plan
- Contingency Plan
- Field Sampling and Analysis Plan
- Health and Safety Plan
- Performance Standard Verification Plan
- Quality Assurance Project Plan
- Design and Construction Quality Assurance Project Plan
- Stormwater Runoff Prevention Plan
- Operation and Maintenance Plan

The reports that will be prepared and submitted during the course of this project include the following:

- Pre-Final Inspection Report
- Construction Completion Report
- Completion of Work Report

1.2 Project Scope of Work

The scope of work for the removal action includes the following activities:

- Clear and grub areas requiring excavation of all trees and brush for disposal off-site.

- Demolish above-grade concrete and metal structures remaining on-site after the Phase I TCR demolition activities in accordance to the design specifications. Sized concrete construction debris will either be used as a sub-base material in areas to be covered with the asphalt cover or will be transported off-site disposal as construction debris. All wood, bricks or metal debris that are removed will be disposed of off-site as construction debris.

- Establish a coordinate grid system along the perimeter of the property outside the fence line and in on-property areas where excavation is required.

- Excavate off-property soils along the western, eastern and southern perimeter of the MMI facility, that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first. XRF screening technology will be used to guide the depth of the excavations during removal.

- Excavate designated on-property soils that are not under concrete or the proposed asphalt cover (including grids I1, J1 and K1 excavated during the Phase I TCR) that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first.

- Conduct confirmatory soil sampling from the excavation floor in grids where the excavation was terminated prior to reaching the historic slag fill material to confirm that all soils that are above the cleanup level have been excavated and removed.

- Backfill all excavated areas once verified to have met the RBRG or have reached historic slag fill, and grading to promote positive drainage in accordance with the design documents. Backfill for areas not covered by asphalt or concrete will be filled with clean imported fill material that has been approved for use based on analytical results and is suitable to maintain vegetative growth.

- Stabilize excavated soils to meet the applicable LDRs for contaminated soils for lead, and any underlying hazardous constituent (UHC) during waste profiling, to render the material nonhazardous for either use as fill in low areas beneath the proposed asphalt cover or for off-site disposal at an approved Subtitle D facility.

- Conduct verification sampling of treated soils using TCLP lead analysis to verify the material has been rendered non-hazardous for lead prior to either placement in low areas beneath the proposed asphalt cover or for off-site disposal as nonhazardous waste.

- Off-site disposal of all treated soils not used to fill low areas beneath the proposed asphalt cover, in accordance with the SOW and the approved design plan.

- Place an asphalt cover over the deteriorated area of the concrete located in southern portion of the site in accordance with the design documents.

- Recondition existing concrete surfaces not under the asphalt cover by sealing any significant cracks and breaks that extend through the concrete surface, followed by encapsulation of the concrete surface, in accordance with the approved design plan.

- Abandon of all existing monitoring wells on site in accordance to applicable State of Ohio regulations (OAC-3745-9-10).

- Remove any existing solid waste including Investigative Derived Waste (IDW) from previous or current removal actions.

- Install a perimeter chain-link fence and three double-swing gates at the completion of the RA to control site access at the site in accordance with the design documents.

- Development of an Operation and Maintenance (O&M) Plan to ensure the integrity of the remedy by maintaining and repairing the concrete and asphalt cover, and the perimeter fencing for a period of thirty (30) years, and as specified in the AOC.

1.3 Summary of Performance Standards (See Table PSVP-I)

The performance standards for the removal action are:

- Lead RBRG for soils: 1,000 mg/Kg total lead on-property and off-property perimeter soils;
- Excavation depth: until either the RBRG of 1,000 mg/Kg total lead is met, or until historic slag fill is encountered, whichever comes first;
- In accordance to the SOW, the more restrictive value of contaminated soil LDR requirements (10 times the Universal Treatment Standard (UTS) or 7.5 mg/L lead) and the nonhazardous characteristic criteria (<5 mg/L TCLP lead) prior to off-site disposal at a permitted Subtitle D landfill;
- Imported Backfill Criteria: TCL/TAL analyses at or below background concentrations, in accordance to the OEPA requirements (refer to Table II, III for complete listing);
- Backfill Procedures: runoff shall be directed to existing catch basins in accordance with the design documents; no erosion of final grade, no ponding;
- XRF calibration for field screening;
- Verification of horizontal and vertical extents of contamination with off-site laboratory confirmatory analyses in grids where excavation was terminated prior to encountering historic slag fill;
- Air monitoring to ensure fugitive dust emissions do not exceed the action levels as specified in the SOW (187.5 µg/m3 for particulate matter);
- O&M Program for a maximum of 30 years, annual inspections and repairs within 30 days; and,
- Site security during the RA, prevent access to the MMI Site and after the RA control access to MMI site.

2.0 EXCAVATION ACTIVITIES

On-property soils identified in the SOW that will not be covered with concrete or the asphalt, and off-site perimeter soils delineated during the Phase II EE/CA, with total lead concentrations greater than the excavation standard of 1,000 mg/Kg will be excavated and staged for treatment. All excavated soils will be treated to meet the more restrictive of the applicable land disposal

restriction (<7.5 mg/L) and nonhazardous characteristic criteria (<5.0 mg/L), for either placement on-site to fill depressions or low areas beneath the asphalt cover or off-site disposal at an approved Subtitle D landfill.

3.0 XRF FIELD SCREENING

An XRF analyzer will be used as a field-screening device to guide the extent and depth of the excavation and assist in determining if the RBRG of 1,000 mg/Kg has been reached prior to encountering the historic slag fill. The XRF instrument will not be used to verify or evaluate the achievement of any performance standard or criteria at the site. XRF screening is discussed in Section 3.0 of the FSAP (Appendix C of the RD/RA Workplan).

Each XRF screening location will be numbered for incorporation into the XRF log-in database as stated in the Field Sampling Plan. The XRF analyzer will be calibrated according to the procedures described in the XRF Standard Operating Procedures presented in Attachment FSAP-1 of the FSAP.

4.0 POST-EXCAVATION SAMPLING

In the event that the results of XRF screening indicate that the in-place soils are below the RBRG prior to reaching the historic slag fill, post-excavation confirmatory samples will be collected. Each confirmatory sample will be collected as a single grab sample from upper 0 to 3 inches of the floor of the sample grid, and thoroughly mixed to achieve a homogenous blend. If the maximum limit of excavation is reached, encountering the historic slag fill material, then confirmatory samples will not be collected. All samples will be submitted to the approved laboratory and analyzed for total lead to confirm that the performance standards have been met and lead-impacted materials have been removed. If the results of confirmation sampling and analysis indicate that the in-place soils did not achieve the excavation performance standard, additional material will be excavated and the process will be repeated until the specified standards are achieved. The extent of additional soil removal will either be based on the results of XRF screening, visual determination, or laboratory analysis throughout the excavation process.

Sampling activities will follow the procedures outlined in the QAPP and the FSAP. All samples will be properly documented and submitted to the off-site laboratory for total lead analysis.

5.0 TREATMENT OF SOIL

Excavated soils will be treated in the treatment containment area. The stabilization process for the lead-impacted soils, sometimes referred to as immobilization or fixation, uses additives to chemically immobilize the hazardous constituents of a contaminated soil by combining the additives and lead-bearing soil within a mixing device. ENTACT has developed a proprietary list of additives for stabilizing heavy metal waste including phosphoric acid, monocalcium phosphate (TSP), monoammonium phosphate, and diammonium phosphate either alone or in combination with Portland Cement.

When applied to lead-impacted soils, the additive/additive blends permit the rapid reaction of free lead with anionic compounds. The first component is a phosphate ion that reacts with metals such as lead to form a salt that is insoluble under normal environmental conditions. The second component is the phosphoric acid buffer system that provides stability to the treated waste mixture under minor environmental changes. The stabilization process and ENTACT patent-pending additives provide the necessary components for successful stabilization of lead contaminated soil with thorough mixing.

A treatability study was completed for the lead-contaminated soils at the MMI site as part of the Phase I TCR action and is included in Appendix E of the RD/RA Workplan.

6.0 TREATMENT VERIFICATION SAMPLING

Verification sampling for the treated soils will follow the sampling protocol outlined in the QAPP and FSAP. The frequency of treatment verification sampling will be one grab sample from every 250 cubic yards of treated material for the first 1,000 cubic yards of material treated, and at increments of 500 cubic yards thereafter.

Verification samples will be submitted to the QAPP-approved laboratory and analyzed for TCLP lead and any underlying hazardous constituent (UHC) identified during the waste profiling, to ensure that the treatment was successful in rendering the material nonhazardous. If the verification sample indicates that treatment has failed to achieve cleanup standards of less the 5.0 mg/L TCLP lead, or the applicable UHC criterion, the entire batch will be retreated and re-sampled.

7.0 TRANSPORTATION AND OFF-SITE DISPOSAL

An estimated 1,800 cubic yards of soils will be excavated and treated as part of the removal action at the MMI site to meet RCRA land disposal restrictions and render the material nonhazardous. The treated material and the stockpiled treated soils from the Holmden Properties RA, not used to fill depressions beneath the asphalt cover system , will be transported off-site to an approved Subtitle D landfill. Each load of treated material/soil that is shipped off-site will be properly tarped and documented by means of the bills of lading completed with each hauling truck leaving the site. These documents will be collected and included in the final report.

8.0 BACKFILL ACTIVITIES

Prior to any excavating or grading activities, ENTACT will install appropriate silt fencing to prevent any erosion and surface runoff as discussed in the Erosion Control Plan (Appendix F of the RD/RA Workplan). After excavation activities have been completed to achieve the performance standards, ENTACT will begin backfilling the RA excavations with clean imported fill suitable for the intended land reuse. ENTACT will grade the excavated areas to ensure proper drainage and to control any additional ponding of water that may occur during implementation of the remedy. Perimeter excavation areas will be planted with new vegetation. Property fencing that is removed to facilitate excavation will also be replaced.

Prior to the backfilling activities, any imported fill material and topsoil will be sampled and analyzed for the 8 RCRA metals, volatile organic compounds (VOCs), pesticides and polychlorinated biphenyls (PCBs), total petroleum hydrocarbons (TPH). In addition, the stockpiled sand fill from on-property areas requiring re-excavation will also be analyzed for the same parameters to ensure the material is suitable for re-use. If TPH levels exceed the OEPA petroleum faction residual saturation concentrations listed in Table I under Ohio Rule 3745-300-8 [8 to 40 mg/Kg for glacial till or silty clay soils] then the backfill shall also be analyzed for the specific semi-volatile organic compounds (SVOCs) required under that rule. Refer to Table II and Table III of the Performance Standard Verification Plan for details on the analytes and compounds to be tested and the associated laboratory method.

One grab sample will be collected for every 10,000 cubic yards of imported fill. Sampling will either be performed by ENTACT or the supplier who will present the necessary documentation that the required testing has been completed and meets the OEPA criteria. All sampling procedures performed by ENTACT will follow protocols outlined in the FSAP and QAPP.

9.0 AIR MONITORING

Throughout the removal action, ENTACT will monitor for fugitive dust emissions form soil excavation, handling and backfilling operations, in accordance with the AOC and SOW. Fugitive particulates at the Property boundary will be monitored in accordance with the FSAP (Appendix C of the RD/RA Workplan)..

Particulate concentrations at the property boundary will not exceed the action levels specified in the SOW: 187.5 μg/m3, which is one half of the 24 hour National Air Quality Standard (NAAQ) for particulate exposure, converted to a one-hour averaging period. Four air monitoring stations will be set up to include, at a minimum, one upwind location and at least two downwind locations. Air monitoring procedures will follow the methodology specified in the FSAP and QAPP.

10.0 LONG-TERM MAINTENANCE

ENTACT will maintain and replace the existing fence at the MMI Site throughout the RD/RA phases, and prevent access and vandalism to the MMI Site. Once the RA has been completed, the perimeter fence will be repaired or replaced as needed as part of the operation and maintenance

procedures to ensure that access to the site is controlled and consistent with future land use at the MMI Site.

Long term maintenance will be conducted in accordance to the O&M Plan, that will provide for long-term integrity of the concrete and asphalt barriers, and the perimeter fence to ensure the integrity of the remedy for a period of 30 years. A Health and Safety Plan will be provided in the O&M for any intrusive construction work that may need to be conducted through the concrete or asphalt covers.

Performance Standard Verification Plan Table PSVP-I Performance Standards
Excavation Activities Excavation of on-property soils in areas not covered by concrete or the asphalt cover or addressed during the Phase I TCR action, and off-site soils delineated in the Phase II EE/CA to either the RBRG of 1,000 mg/Kg total lead is achieved or the historic slag fill is encountered, whichever comes first. Excavated soils will be staged for treatment in the pre-treatment staging area.
XRF Field Screening The XRF instrument will be used only as a field-screening tool to assist in determining if the performance standard of 1,000 mg/Kg has been achieved prior to reaching the historic slag fill. The XRF instrument is used only to guide the extents of excavation and not to verify or evaluate the achievement of any performance standard or criteria at the site. Calibration will be in accordance to the Field Sampling Plan. XRF will screen each excavated grid square in four locations. If XRF screening indicates that the criteria has been met before the historic slag is encountered, then a confirmatory sample will be collected for fixed laboratory analysis to verify that the performance standard has been met as described below.
Post-Excavation Sampling For grids where excavation is terminated before the historic slag is encountered, a grab sample will be collected from the upper 0 to 3 inches in the center of each grid, and submitted to the approved laboratory for analysis of total lead. The sampling procedures will follow the approved QAPP and Field Sampling Plan
Treatment Soils will be treated in batches within the constructed treatment containment area using a pre-determined ratio of additive blend to impacted soils to effectively render the soils nonhazardous (< 5.0 mg/L lead). Treatment to the 5.0 mg/L lead criteria is below the RCRA LDR for lead in contaminated soils of 7.5 mg/L (10 times the UTS of 0.75 mg/L). Treated soils will be disposed off-site at an approved Subtitle D landfill..
Verification Sampling Verification sampling for the treated soils will be conducted prior to placement and consolidation in order to verify that the treatment standard of 5.0 mg/L lead has been met. The samples will consist of one grab sample for every 250 cubic yards for the first 1,000 cubic yards treated, and at increments of one sample for every 500 cubic yards thereafter in accordance to the approved QAPP and FSAP.
Asphalt Cover A minimum 4-inch asphalt cover will be placed over the southern portion of the site where the concrete has deteriorated. The asphalt cover will be placed in accordance with the approved design specifications.

Performance Standard Verification Plan Table PSVP-I continued Performance Standards
Backfill Activities All excavations will be backfilled with clean fill material. One
representative sample will be collected per source area for each 10,000 cubic yards used.
The backfill sample will be analyzed for the following parameters: 8 RCRA Metals,
Volatile Organic Compounds, pesticides, PCBs and TPH to ensure the Ohio EPA standards
for clean fill are met. Samples procedures will follow protocols outlined in the QAPP and FSAP
The backfill will be properly graded to ensure proper drainage and to avoid ponding. Off-site
perimeter areas that are backfilled will be restored with vegetation.
Transportation and Off-Site Disposal Treated soils that have been verified to be rendered
nonhazardous will be transported off-site to an approved Subtitle D landfill. All material will be
classified as Class II Non-Hazardous Waste and will meet the applicable RCRA
LDRs. Each truck load will be properly documented by means of the bills of lading
All IDW generated during this and previous investigations will be properly disposed of.
Long-Term Maintenance Annual inspections to ensure access to site is
controlled and consistent with future land use at the MMI site. Implementation of operation
and maintenance plan to maintain the concrete and asphalt cover for a period of 30 years.

Performance Standard Verification Plan Table PSVP-II Target Compound List (TCL)			
	METHOD REPORTING LIMITS		
VOLATILES	Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg
Chloromethane	5	5	1200
Bromomethane	5	5	1200
Trichlorofluromethane	5	5	1200
Dichlorodifluromethane	5	5	1200
Vinyl chloride	5	5	1200
Chloroethane	5	5	1200
Acetone	25	25	1200
Carbon disulfide	5	5	1200
1,1-Dichloroethene	5	5	1200
1,1-Dichloroethane	5	5	1200
1,2-Dichloroethene (cis / trans))	5	5	1200
Chloroform	5	5	1200
1,2-Dichloroethane	5	5	1200
2-Butanone	25	25	1200
1,1,1-Trichloroethane	5	5	1200
Carbon tetrachloride	5	5	1200
1,1-Dichloropropene	5	5	1200
Bromochloromethane	5	5	1200
Bromodichloromethane	5	5	1200
1,2-Dibromomethane	5	5	1200
1,2-Dichloropropane	5	5	1200
1,3-Dichloropropane	5	5	1200
2,2-Dichloropropane	5	5	1200
1,3-Dichloropropene (cis, trans)	5	5	1200
Trichloroethene	5	5	1200
Dibromomethane	5	5	1200
Dibromochloromethane	5	5	1200
1,1,2-Trichloroethane	5	5	1200
Benzene	5	5	1200
tert-Butylbenzene	5	5	1200
sec-Butylbenzene	5	5	1200
n-Butylbenzene	5	5	1200
trans-1,3-Dichloropropene	5	5	1200
1,2,3-Trichloropropane	5	5	1200
1,2-Dibromo-3-chloropropane	5	5	1200
Isopropylbenzene	5	5	1200
n-Propylbenzene	5	5	1200
1,2,4-Trimethylbenzene	5	5	1200
1,3,5-Trimethylbenzene	5	5	1200

Performance Standard Verification Plan Table PSVP-II Target Compound List (TCL)			
	METHOD REPORTING LIMITS		
VOLATILES	Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg
Bromoform	10	5	1200
Bromobenzene	10	5	1200
4-Methyl-2-pentanone	25	25	1200
Methyl-tert-butyl-ether	10	5	1200
2-Hexanone	25	25	1200
Tetrachloroethene	10	5	1200
Toluene	10	5	1200
1,1,2,2-Tetrachloroethane	10	5	1200
1,1,1,2-Tetrachloroethane	10	5	1200
Naphthalene	10	5	1200
Chlorobenzene	10	5	1200
1,2-Dichlorobenzene	10	5	1200
1,3-Dichlorobenzene	10	5	1200
1,4-Dichlorobenzene	10	5	1200
1,2,3-Trichlorobenzene	10	5	1200
1,2,4-Trichlorobenzene	10	5	1200
Hexachlorobutadiene	10	5	1200
2-Chlorotoluene	10	5	1200
4-Chlorotoluene	10	5	1200
p-Isopropyltoluene	10	5	1200
Ethyl benzene	10	5	1200
Styrene	10	5	1200
Xylenes (total)	10	5	1200

Performance Standard Verification Plan Table PSVP- II continued Target Compound List (TCL)			
	METHOD REPORTING LIMITS		
SEMIVOLATILES (Method 8270)	Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg
2,4-Dinitrophenol	25	1650	25000
4-Nitrophenol	5	330	25000
Dibenzofuran	5	330	25000
2,4-Dinitrotoluene	5	330	25000
Diethylphthalate	5	330	25000
4-Chlorophenyl-phenyl ether	5	330	25000
Flourene	5	330	25000
4-Nitroaniline	5	330	25000
4,6-Dinitro-2-methylphenol	5	330	25000
N-nitrosodiphenylamine	25	1650	25000
4-Bromophenyl-phenylether	5	330	25000
Hexachlorobenzene	5	330	25000
Pentachlorophenol	5	330	25000
Phenanthrene	5	330	25000
Anthracene	5	330	25000
Carbazole	5	330	25000
Di-n-butylphthalate	5	330	25000
Fluoranthene	5	330	25000
Pyrene	5	330	25000
Butylbenzylphthalate	5	330	25000
3,3-Dichlorobenzidine	25	1650	25000
Benzo(a)anthracene	5	330	25000
Chrysene	5	330	25000
bis(2-Ethylhexyl)phthalate	5	330	25000
Di-n-octylphthalate	5	330	25000
Benzo(b)fluoranthene	5	330	25000
Benzo(k)fluoranthene	5	330	25000
Benzo(a)pyrene	5	330	25000
Indeno(1,2,3-cd)pyrene	5	330	25000
Dibenzo(a,h,i)anthracene	5	330	25000
Benzo(g,h,i)perylene	5	330	25000

Performance Standard Verification Plan Table PSVP-II continued Target Compound List (TCL)			
	METHOD REPORTING LIMITS		
SEMIVOLATILES	Water µg/L	Low Soil µg/Kg	Med. Soil µg/Kg
Phenol	5	330	10000
bis(2-Chloroethyl) ether	5	330	10000
2-Chlorophenol	5	330	10000
1,3-Dichlorobenzene	5	330	10000
1,4-Dichlorobenzene	5	330	10000
1,2-Dichlorobenzene	5	330	10000
2-Methylphenol	5	330	10000
2-Methyl-4,6-dinnitrophenol	25	1650	10000
4-Methylphenol	5	330	10000
N-Nitroso-di-n-propylamine	25	1650	10000
Hexachloroethane	5	330	10000
Nitrobenzene	5	330	10000
Isophorone	5	330	10000
2-Nitrophenol	5	330	10000
2,4-Dimethylphenol	5	330	10000
bis(2-Chloroethoxy) methane	5	330	10000
2,4-Dichlorophenol	5	330	10000
1,2,4-Trichlorobenzene	5	330	10000
Naphthalene	5	330	10000
4-Chloroaniline	5	330	10000
Hexachlorobutadiene	5	330	10000
4-Chloro-3-methylphenol	5	330	10000
2-Methylnaphthalene	5	330	10000
Hexachlorocyclopentadiene	5	330	10000
2,4,6-Trichlorophenol	5	330	10000
2,4,5-Trichlorophenol	5	330	25000
2-Chloroaphthalene	5	330	10000
2-Nitroaniline	5	330	25000
Dimethyl phthalate	5	330	10000
Acenaphthylene	5	330	10000
2,6-Dinitrotoluene	5	330	10000
3-Nitroaniline	5	330	25000
Acenaphthene	5	330	10000

Performance Standard Verification Plan Table PSVP-II continued Target Compound List (TCL)		
	METHOD REPORTING LIMITS	
PESTICIDES/AROCLORS	Water µg/L	Soil µg/Kg
α-BHC	0.05	0.05
β-BHC	0.05	0.05
δ-BHC	0.05	0.05
γ-BHC (Lindane)	0.05	1.7
Heptachlor	0.05	1.7
Aldrin	0.05	0.05
Heptachlor epoxide	0.05	1.7
Endosulfan I	0.05	1.7
Dieldrin	0.1	3.3
4,4-DDE	0.1	3.3
Endrin	0.1	3.3
Endosulfan II	0.1	3.3
4,4-DDD	0.1	3.3
Endolsulfan sulfate	0.1	3.3
4,4-DDT	0.1	3.3
Methoxychlor	0.5	17.0
Endrin aldehyde	0.1	3.3
Chlordane	2.5	85.0
Toxaphene	2.5	85.0
Aroclor-1016	1.5	33.0
Aroclor-1221	1.5	33.0
Aroclor-1232	1.5	33.0
Aroclor-1242	1.5	33.0
Aroclor-1248	1.5	33.0
Aroclor-1254	1.5	33.0
Aroclor-1260	1.5	33.0

Performance Standard Verification Plan Table PSVP-III 8 RCRA Metals List	
ANALYTE	**DETECTION LIMIT (mg/L)**
Arsenic	1
Barium	10
Cadmium	0.05
Chromium	1
Lead	1
Mercury	0.1
Selenium	0.5
Silver	1

APPENDIX C

FINAL FIELD SAMPLING & ANALYSIS PLAN

Appendix C
Final Field Sampling and Analysis Plan
Master Metals, Inc. Site
Cleveland, Ohio

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Appendix C
Field Sampling and Analysis Plan
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1.0 INTRODUCTION

This Field Sampling and Analysis Plan (FSAP) supplements the Quality Assurance Project Plan (QAPP) to the Master Metals, Inc. (MMI) Site Removal Design and Removal Action (RD/RA) Work Plan. This FSAP describes the procedures to be used for collection of samples, including soil, treated material, waste and air samples.

1.1 Site Background

The MMI Superfund Site (the "Site') covered under the AOC includes the former MMI lead facility (the "Facility") located at 2850 West Third Street, Cleveland, Cuyahoga County, Ohio and the stockpiled treated soils removed from the surrounding residential property at 1157, 1159 and 1167 Holmden Avenue (the "Holmden Properties") where lead-impacted material from Master Metals was deposited as fill (USEPA, 1999). The Site is situated in Township 7 North, Range 12 West, Section 17, ¼ NE, ¼ SW, ¼ SW, with coordinates obtained from the Facility Index System (FINDS) listed as 41 degrees, 28 minutes, 26 seconds latitude and -81 degrees, 40 minutes, 31 seconds longitude. The site location is illustrated in Figure 1-1 of the RD/RA Workplan.

The MMI property is a triangular-shaped parcel encompassing approximately 4.3 acres in the "flats" area of downtown Cleveland, a heavily industrialized sector of the city. The site is bordered on west by rail yards owned by the Baltimore & Ohio (B&O) Railroad, the east by West Third Street and B&O railroad tracks and on the south by a dead-end road and an abandoned industrial property. LTV Steel owns the property to the south and north. The Cuyahoga River is located approximately 1,250 feet east of the facility and flows north toward Lake Erie (ENTACT, 1999). An athletic field and playground are situated approximately 1,000 feet to the west. The nearest residential property to the former facility is approximately 2,000 feet to the northwest (USEPA, 1999).

Major site features, prior to a 1997-1998 time-critical removal (TCR) action, included an office building, a secondary lead smelting furnace building, two large brick baghouses, the roundhouse building, storage buildings, material storage bins and boxes, and an above-ground storage tank farm (ENTACT, 1998). All buildings, except for the roundhouse and the attached office building in the northern corner of the property, were razed as part of the Phase I TCR (ENTACT, 1998) and all remaining feedstock and debris materials were decontaminated and/or treated and disposed of off-site as either special waste or as hazardous waste (ENTACT, 1998). The MMI facility property is currently vacant with the exception of the roundhouse, and the majority of the open land surface covered with concrete or asphalt except along the site boundaries. Current site features are illustrated in Figure 1-2 of the RD/RA Workplan.

Stormwater drainage is directed toward one of five on-site stormwater catch basins that connect to the combined sewer system operated by the Northeast Ohio Regional Sewer District (NEORSD) (ESC, 1991). Locations of the sewers are illustrated on Figure 4-1 of the RD/RA Workplan. Topographic maps suggest that the direction of groundwater flow and surface water flow in the vicinity of MMI is to the northeast toward the Cuyahoga River (ENTACT, 1999).

The MMI facility was constructed in 1932 on slag fill by National Lead Industries, Inc. (NL) who owned and operated the facility as a secondary lead smelter, producing lead alloys from lead-bearing dross and scrap materials. NL Industries also engaged in battery cracking operations at this facility. In 1979, the facility was purchased from NL Industries by Master Metals who continued to run secondary lead smelter operations (USEPA, 2001a).

As part of their operations, the Master Metals facility received lead-bearing materials classified and regulated under Resource Conservation and Recovery Act (RCRA) as D008 hazardous waste from off-site sources (USEPA, 2001a). This waste was converted into lead ingots using pot and rotary furnaces equipped with baghouses to collect particulate matter from the furnace that consisted predominantly of lead dust. The sludge that accumulated in the furnaces after smelting was classified as K069 waste hazardous waste. Finished lead ingots were stored in a roundhouse at the north end of the property prior to shipment off-site.

Based on background information, the by-products produced from smelting operations included furnace flux, slag, dross, baghouse fines and furnace sludge (USEPA, 2001a). With the exception of slag, which was tested and disposed of off-site, most of the lead-bearing by-products were recycled back into the furnace. Cooling water used in the operations was diverted to a combined sewer system operated by the NEORD (ESC, 1991).

Violations relating to noncompliance and poor operating practices are documented in various state and federal agency reports, summarized in the Section III of the AOC, presented in Appendix A of the RD/RA Workplan and summarized in Section 1.3.1 of the RD/RA Workplan. On August 5, 1993, as a result of continuing RCRA violations, the Ohio EPA Director ordered MMI to cease operating the facility until it could demonstrate compliance (USEPA, 2001a). Operations never did resume at the MMI facility and Bank One of Ohio took possession of all MMI cash collateral and accounts receivable. The current property owner remains MMI. The former facility president, Mr. Douglas Mickey, is deceased (USEPA, 2001).

1.2 Past Data Collection Activities

Numerous investigations have been conducted by MMI at the facility between 1990 to 1998 to determine the nature and extent of constituents of concern related to former operations.

1.2.1 Compliance Technologies, December 1990

Compliance Technologies, Inc. (CTI) conducted a Phase II environmental assessment of the MMI site from December 3 through December 11, 1990. The investigation included the advancement of 31 soil borings to a maximum depth of 10 feet, and the installation of four monitoring wells to a depth of 15 feet. The purpose of the investigation was to evaluate subsurface and groundwater conditions beneath the MMI facility and determine the impact of prior slag disposal/landfill activities on these media (CTI, 1991b).

Forty-four subsurface soil samples were collected from the 31 borings located in or near the MMI facility. The samples were collected from depths ranging between two to ten feet below

ground surface (CTI, 1991b). The soil samples were submitted to BHM Analytical Laboratory, Chagrin Falls, Ohio and analyzed for the eight RCRA metals (i.e. arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver). The analytical results showed on-site lead concentrations ranging from 18.1mg/Kg to 14, 070 mg/Kg, with lead levels one to two orders of magnitude above the other metals detected. Off-site concentrations of lead in subsurface samples ranged from 7.85 to 55 mg/Kg. Slightly elevated concentrations of chromium and cadmium were observed in only 17 of the 44 samples. Historical sample locations and the associated lead concentrations are shown in Figure FSAP-1.

Based on boring logs, saturated conditions were reported to be present between eight to ten feet below ground surface across the facility. Four groundwater samples were collected from the newly-installed monitoring wells on December 28, 1990 using hand bailers and were not filtered. Total lead concentrations ranged between 0.45 mg/L to 1.39 mg/L.

In addition to the soil samples, two samples were collected of the brick and slag material and analyzed for the TCLP 8 RCRA Metals, reactive sulfide, total cyanide, pH and flash point to determine if these materials were hazardous by characteristic (CTI, 1991b). Lead was present in the slag material at 7,075 mg/Kg with leachable lead detected in the slag material at 16.1 mg/L.

1.2.2 Ecology & Environment, July 1992

On July 14, 1992, Ecology and Environment (E&E), on behalf of the U.S. EPA, collected seven surface samples on-site (SS1 - SS7) and three off-site surface soil samples from outside the fence to the east, south and west (SS8 – SS10) as part of a site assessment and hazard evaluation of the MMI facility. All soil samples were submitted to American Environmental Laboratories, Inc. of Bedford, Ohio for analysis of the eight RCRA metals.

Lead concentrations in the on-site surface soil samples ranged from 6,020 to 115,000 mg/Kg. Off-site surface soil samples collected outside the fence showed lead concentrations ranging between 24,000 to 43,100 mg/Kg (E&E, 1992). Sample locations and the associated lead levels are presented in Figure FSAP-1. Once again, lead values were 1-2 orders of magnitude higher than the seven other metals. Some results exhibited minor arsenic, barium, cadmium, and chromium concentrations, relative to the co-located lead concentrations (E&E, 1992).

In July 1992, E&E, on behalf of U.S. EPA, collected samples proximate to the facility property to determine if the facility contaminants were subject to airborne transport. Analysis of these samples (SS8 - SS10) for RCRA metals showed total lead levels of 6,020 - 43,100 ppm. Sample locations and analytical results are illustrated in Figure FSAP-1.

1.2.3 Phase I Time Critical Removal

A Phase I TCR was performed at the Site by ENTACT between June 9, 1997 and January 6, 1998 in accordance with the terms of the AOC Docket number V-W-97-C-402, issued April 17, 1997 by the USEPA Region 5. As part of the time-critical removal, all exposed on-site surface areas (e.g., not covered by concrete) were excavated to a maximum depth of two feet or until

slag fill material (e.g., slag, cinders, etc.) were encountered. XRF information collected from the excavations exhibited lead concentrations up to 39,000 ppm in the remaining slag fill material.

1.2.4 Phase II Engineering Evaluation and Cost Assessment (EE/CA)

A five-step Phase II EE/CA investigation was conducted by ENTACT in 1998 in accordance with the terms of the AOC Docket number V-W-97-C-402, issued April 17, 1997 by the USEPA Region 5. On-site soil sampling included the advancement of seven borings. Results indicated that 5 of the 7 borings exceeded 1,500-mg/Kg lead at total depth. Historic slag was encountered at approximately three to four feet which is consistent with the information collected during the Phase I TCR (ENTACT, 1998b. The soil sampling locations are illustrated in Figure 1-3 of the RD/RA Workplan. The on-site sampling indicated that significant lead concentrations, up to 35,000 mg/Kg, remained in on-site soils to a depth of 3 to 4 feet. These areas were either covered with the existing concrete surface or had been excavated and backfilled with 2 feet of clean fill as part of the Phase I TCR. Therefore, in areas where the concrete was competent and in uncovered areas that were excavated as part of the Phase I TCR, the potential for further entrainment of airborne lead had been mitigated and was no longer considered a concern (ENTACT, 1998b). However, a potential for airborne lead releases did exist in areas where the concrete was compromised. These areas were recommended for repair to mitigate this airborne migration route (ENTACT, 1998b).

An off-site perimeter surface soil survey was conducted adjacent to the fence line along the western, eastern and southern boundaries of the MMI facility property using an XRF instrument. Samples were collected at nineteen locations designated in Figure 1-3 of the RD/RA Workplan. Results of the perimeter lead survey showed lead levels ranging from 931 ppm to 36,587 ppm within the upper 12 to 24 inches of soils, decreasing rapidly with depth. The EE/CA found that the surficial elevated lead levels continue to pose a potential ingestion or inhalation threat, and recommended that additional removal action be conducted in these areas (ENTACT, 1998b).

Off-site sampling included the collection of nine off-site surface soil samples along Quigley Avenue. The results showed levels of the average lead concentration to be below the Superfund residential soil screening level of 400 mg/Kg. No further action was recommended (ENTACT, 1998b).

Groundwater sampling conducted in 1991 showed total lead concentrations ranging from 0.45 mg/L to 1.35 mg/L, total chromium concentrations ranging from 0.02 mg/L to 1.33 mg/L and lesser concentrations of arsenic and cadmium (CTI, 1991). Groundwater sampling of the three remaining monitoring wells during the 1998 EE/CA investigation showed the presence of lead, arsenic, cadmium and chromium at levels that have either remained at, or have declined from, the 1991 sampling results. Groundwater is not used as a source of drinking water within a four-mile radius of the site, with Lake Erie supplying the greater Cleveland area with its drinking water supply. Based on the low concentrations of metals in the groundwater and the lack of any potential downgradient receptors, the groundwater migration pathway was eliminated as a concern (ENTACT, 1998b).

The EE/CA assessment verified that lead was the predominant hazardous constituent of concern at the site, with lesser occurrences of arsenic. Removal action directed at lead exceedences would also

address the co-located elevated levels of arsenic. Based on a streamlined risk evaluation, a risk-based remediation goal (RBRG) for lead of 1,000 mg/Kg was established for on-site and off-site perimeter soils (ENTACT, 1998b). This final removal action has been designed to address the remaining lead impacts defined in the EE/CA and associated with former facility operations.

1.3 Statement of Objectives

In accordance with the AOC and Site Statement of Work (SOW), presented in Appendix A of the RD/RA Workplan, the following remedial actions will be accomplished during RA:

- Clear and grub areas requiring excavation of all trees and brush for disposal off-site.

- Demolish all above-grade concrete structures remaining on-site after the Phase I TCR demolition activities in accordance to the design specifications. Concrete construction debris will either be used as a sub-base material in areas to be covered with asphalt or will be transported off-site disposal as construction debris. All wood or metal debris will be disposed of off-site.

- Establish a coordinate grid system along the perimeter of the property outside the fence line and in on-property areas where excavation is required.

- Excavation of lead-impacted on-property soils that are not under concrete or the proposed asphalt cover area, including grid areas excavated during the Phase I TCR but identified in the SOW, that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first.

- Excavation of off-property soils along the western, eastern and southern perimeter of the MMI facility, that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first. XRF screening technology will be used to guide depth of the excavations during removal.

- Confirmatory soil sampling from the excavation floor in grids where the excavation was terminated prior to reaching the historic slag fill material to confirm that all soils that are above the cleanup level have been excavated and removed.

- Backfill all excavated areas either determined to have met the RBRG, or have reached historic slag fill, with clean imported fill material that has been approved for use based on analytical results.

- Stabilization of excavated soils to meet the applicable LDRs for contaminated soils for lead and any underlying hazardous constituent (UHC) to render the material nonhazardous for either use as fill in low areas beneath the proposed asphalt cover areas or for off-site disposal at an approved Subtitle D facility.

- Verification sampling of treated soils using TCLP lead analysis to verify the material has been

rendered non-hazardous for lead prior to either placement in low areas beneath the proposed asphalt cover or for off-site disposal as nonhazardous waste.

- Off-site disposal of all treated soils, including stockpiled soils from the Holmden Properties Removal Action, not used to fill low areas beneath the proposed asphalt cover, in accordance with the SOW and the approved design plan.

- Placing an asphalt cover over the deteriorated area of the concrete located in southern portion of the site as delineated in the design documents.

- Recondition existing concrete surfaces not under the asphalt cover by sealing any significant cracks and breaks that extend through the concrete surface, followed by encapsulation of the concrete surface, in accordance with the SOW and approved design plan.

- Abandonment of all existing monitoring wells on site in accordance to applicable State of Ohio regulations (OAC-3745-9-10).

- Removal of any existing solid waste including Investigative Derived Waste (IDW) from previous or current removal actions.

- Applying clean fill in areas addressed during the Phase I TCR along the western perimeter that are currently below grade, and appropriately grading these areas to prevent run-on to the site or run-off from the site.

- Installation of perimeter chain-link fence at the completion of the RA to control site access at the site.

- Perform Operation and Maintenance activities to ensure the integrity of the remedy by maintaining and repairing the concrete and asphalt cover, and the perimeter fencing for a period of thirty (30) years, as required under CERCLA.

1.4 Sampling Activities

This FSAP for the Remedial Action at the MMI Site will be implemented for the following types of samples:

- Air Samples
- Excavation Confirmatory Soil Samples
- X-Ray Fluorescence Field Screening Samples
- Treated Material Verification Samples
- Imported Backfill Characterization Samples

The objectives of these sampling activities include:

- Direct removal action tasks;

- Verification of that removal criteria have been achieved;
- Verification of treated soils for on-site placement; and
- Collection of data to determine if implementation of the Contingency Plan is necessary due to constituent concentrations in air or surface water.

2.0 FIELD SAMPLING ACTIVITIES IMPLEMENTATION

2.1 Establishment of Coordinate Grid System

A coordinate grid system (CGS) will be established in order to provide a coordinate system for tracking sampling and excavation activity in the field. The approximate location of the CGS for the impacted area to be delineated is presented on Figure FSAP-2. The CGS will employ square grids of 50 feet by 50 feet superimposed completely over the impacted area. Actual marking of grid corners in the field will be made by wooden stakes and/or spray paint. ENTACT will install wooden stakes or metal posts to delineate benchmarks so that the grids can be easily relocated in the field should remedial activities disrupt grid markings or stakes. This coordinate system will be used to provide reference markers for 1) confirmatory soil sampling, and 2) XRF field-screening activities.

2.2 Sample Identification System

A sample identification system will be implemented in order to properly track sampling activities. The sampling activities and examples of the identification coding system associated with each type are listed below with a following explanation:

Samples Type	Identification System
Air Samples:	
TSP High Volume Samples	TSP-Unit#-001
Personal/Area Low Volume Samples	PAS-Unit#-001
Soil Samples:	
X-Ray Fluorescence Field Screening	X-01-1
Post-Excavation Confirmatory Samples	V-01-2.0'
Treated Material-Confirmation (TCLP) Samples	TS-001
Imported Backfill Samples	BF-001
Waste Characterization Samples:	
Solid Waste (general waste and stabilized soils if off-site disposal is needed)	W-001
Wastewater (not used for dust control measures)	WW-001

Quality Control Samples:	
Field Duplicate Samples for Soil, Treated Material	V-01-2.0' D TS-001D
Field Rinsate Blanks	FB-001

All numbering sequences shown above with "001" will begin with the number "001" and will continue sequentially (i.e., FB-001, FB-002, etc.; TSP-1-001, TSP-1-002, etc.) until the final samples for the removal action are collected. Air monitoring samples will include the type and station number to identify which air station the measurement was recorded on. For example, TSP-2-002 will indicate the second measurement on TSP Station No. 2.

X-Ray Fluorescence (XRF) excavation samples will be numbered for incorporation into the XRF log-in database. The samples will be numbered with the grid identification number and the specific screening location within the grid. The grid numbering system is explained in the following section. For example, an XRF sample obtained from an excavation in Grid 1, from the third location out of four within the grid, will be designated X-01-3. For a reverification of the same location, a, b, c, etc will follow the sample identification code.

If excavation is terminated prior to reaching the historic slag fill, a confirmatory soil sample will be collected to verify that the RBRG has been achieved. These confirmation samples to be sent to the approved laboratory will be obtained as a single grab sample from within that grid. Each soil sample will be numbered with the unique grid identification number and the sampling depth from ground surface. For example, a post-excavation confirmation sample obtained from the excavation in Grid 1 at 2 feet below original grade, will be designated V-01-2.0'

3.0 SAMPLING PROCEDURES

3.1 XRF Field Screening

The purpose of the XRF field screening is to guide the extent of the excavation until either the RBRG is achieved or until historic slag is encountered. The XRF data will not be used to verify or evaluate the achievement of any performance standard or criteria at the site. XRF screening will only be used to assist in conducting the removal activities at the site.

The following procedures describe the overall sampling process during the XRF field screening.

a. The sampling team will adhere to the Health and Safety Plan requirements.

b. The XRF will be used to obtain measurements at four locations per each grid designated for excavation in on-foot increments. These four locations will be determined based on spatial distribution, or visual observations.

c. If the XRF results indicate that the lead is below 1,000 mg/Kg total lead before the historic slag is encountered, excavation will be terminated and a confirmatory sample will be collected to verify that the action level has been achieved for that grid as described in Section 3.2 of the FSAP.

d.. If XRF screening results reveal lead-impacted material above the clean-up criteria of 1,000 mg/Kg, then excavation will continue in one-foot increments with XRF screening conducted at each one-foot increment until the maximum depth interval is reached (i.e., when the historic slag is encountered, typically between 3 and 4 feet below ground surface)

XRF Field Screening Methodology

ENTACT's XRF Standard Operating Procedures (SOP) are presented in Attachment FSAP-1 of the FSAP. XRF analysis for total lead on soil and solid media will be performed as follows:

a. Sampling teams will adhere to the Health and Safety Plan requirements.

b. An approximate 6-inch by 6-inch square area on the excavation floor will be cleared of any stones or debris and flattened with a trowel, with care being taken to remove as little surficial soil as possible, to provide a flat area for XRF analysis as described in Section XIII (A) of the XRF SOP.

c. The XRF probe will be placed on the flat, compacted soil surface, activated and held in place for the 60-second scanning period.

e. One measurement will be collected in each grid quadrant, and these readings will be written into the field logbook. If any of the four XRF readings collected in the grid indicate that the

lead is present above 1,000 mg/Kg, excavation across the entire grid will continue.

f. The sample identification number for each measurement will be entered into the unit's computer memory and saved along with the result. The data will then be downloaded onto a computer hard disk file at end of each day. The results will also be written into the field logbook.

3.2 Post-Excavation Confirmatory Soil Sampling

If the excavation was terminated before historic slag was encountered, ENTACT will collect one (1) grab confirmatory soil sample. One grab sample will be collected from the excavation floor in each grid at a depth of 0 to 3 inches. If the historic slag is encountered prior to achieving the RBRG of 1,000-mg/Kg lead, the excavation will be terminated and no confirmatory sample will need to be collected from the excavation floor.

All confirmatory samples will be submitted to the approved laboratory for total lead analysis by EPA Method 6010B. Analytical parameters and test methods are presented on Table FSAP-1. Post-excavation confirmatory soil samples to be submitted for laboratory analysis will be performed as follows:

a. The sampling team will adhere to the Health and Safety Plan requirements.

b. Designated sampling locations will be identified. Photographs will be maintained to document sample locations.

c. Staging areas for sample collection will be established. Clean, plastic holding containers will be placed adjacent to the areas to be sampled during sample collection. The following tools and supplies will be prepared for use:

- Field Logbook;
- Plastic or glass laboratory-supplied sample containers;
- Stainless steel or plastic disposable trowels;
- Zip-Lock bag or equivalent sample bags or stainless steel bowl;
- Measuring tape;
- Paper plates;
- Distilled water, low-phosphate detergent, and brushes;
- Disposable gloves;
- Trash bags; and
- Three 5-gallon buckets to carry equipment and for decontamination liquids if reusable sampling equipment is used.

d. A sufficient amount of soil will be retrieved by sample trowel, placed into a clean Zip-lock bag or stainless bowl and mixed to achieve a homogeneous sample then transferred to the sample containers.

e. Field notes will be completed and will include identification of the soil sample number, time and date of collection, color, and brief description.

f. Chain-of-custody documents will be prepared according to procedures outlined in Sections 4.1.2.4. and 5.0 of the QAPP. Sample containers will be labeled in accordance with the predetermined sample numbering system, and sealed in a plastic bag for shipment to the laboratory for analysis.

g. All reusable sampling equipment will be decontaminated utilizing a detergent wash and potable water rinse, followed by a distilled water rinse and drying with disposable towels between each sampling event. All disposable sampling media will be placed into designated site containers.

3.3 Treatment Confirmation Samples

Treated material will be sampled and analyzed for toxicity characteristic leaching procedure (TCLP) lead and any identified underlying hazardous constituent (UHC). The frequency of sampling will be one (1) grab sample from every 250 cubic yards of treated material for the first 1,000 cubic yards, then one grab sample for every 500 cubic yards, thereafter. Soils that exhibit the toxicity characteristic for lead will be treated to render the waste non-hazardous i.e., less than 5.0 mg/L TCLP lead and meet the LDR of < 7.5 mg/L TCLP lead. The LDRs will also be met of any underlying UHC. Soils will be sampled and analyzed for TCLP lead by EPA Method 1311/6010B or 1311/6020. Analytical parameters and test methods are shown on Table FSAP-1. The following field methods will be utilized for these sampling efforts:

a. The sampling team will adhere to the Health and Safety Plan requirements.

b. A sufficient amount of material will be retrieved by sample trowel and placed into a clean, stainless steel or plastic bowl or Zip-Lock bag or equivalent and mixed well. The sample will then be inserted into the sample containers.

c. Field notes will be completed and will include identification and storage location of the batch being sampled, sample number, data and other pertinent information.

d. Chain-of-custody documents will be prepared, sample containers will be labeled in accordance with the predetermined identification system and samples will be sealed and shipped to the laboratory for analysis.

e. All re-usable sampling equipment will be decontaminated utilizing a detergent wash and potable water rinse, followed by a distilled water rinse and drying with disposable towels between each sampling event. All disposable sampling media will be placed into designated waste containers.

3.4 Sampling of Off-site Fill Materials

Samples of fill material brought in from offsite sources will be collected according to the following procedures. Initially, each source of fill material will be sampled once for the eight RCRA metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver), volatile organic compounds (VOCs), pesticides/PCBs, and total petroleum hydrocarbons (TPH). If TPH levels exceed the OEPA petroleum faction residual saturation concentrations listed in Table I under Ohio Rule 3745-300-8 (8 to 40 mg/Kg for glacial till or silty clay soils), the fill will be sampled for semi-volatile organic compounds (SVOCs). The VOC sample will be collected as a grab sample and will not be homogenized. The remaining sample parameters will be collected from a composite sample of at least four aliquots obtained from the source area or stockpiled material from the source area. Sample test methods and sample container requirements are listed on Table FSAP-1. The source location of the backfill material will be documented by source location and address. Sampling will then be performed on every additional 10,000 cubic yards from the same source area by grab sample for total lead. The backfill samples will be collected as follows:

a. The sampling team will adhere to the Health and Safety Plan requirements.

b. A sufficient amount of material will be retrieved by sample trowel and placed into a clean, stainless steel or plastic bowl or Zip-Lock bag or equivalent and mixed well. The sample will then be inserted into the sample containers.

c. Field notes will be completed and will include identification and storage location of the batch being sampled, sample number, date and time collected and other pertinent information.

d. Chain-of-custody documents will be prepared, sample containers will be labeled in accordance with the predetermined identification system and samples will be sealed and shipped to the laboratory for analysis.

e. All sampling equipment will be decontaminated utilizing a detergent wash and potable water rinse, followed by a distilled water rinse and drying with disposable towels between each sampling event. All disposable sampling media will be placed into designated site containe

4.0 AIR SAMPLING AND ANALYSIS PLAN

Air monitoring will be conducted on site to ensure that all personnel and local residents are not exposed to levels of particulate matter or airborne lead concentrations in excess of the regulated limits, and to ensure that contaminants are not migrating off site. For this project, Clean Air Act monitoring methodologies will be employed to monitor for respirable dust and lead emissions in addition to the OSHA defined air monitoring. Air sampling equipment to be used as part of the removal action include the use of perimeter high volume monitors (i.e., Total Suspended Particulate (TSP) and low volume personal/area air monitors. Standard operating procedures for the air samples used is presented in Appendices FSAP-2, and FSAP-3. The air-monitoring program designed to protect worker safety is detailed in Section 7.0 of the Health and Safety Plan.

Two types of air samples will be used at the site, and analyzed at the laboratory. These consist of high volume total suspended particulate air sampler for analysis of total particulate and total lead. A list of the sample parameters and test methods are presented on Table FSAP-1. Baseline air monitoring will begin one week prior to the initiation of the removal action and will be conducted on a regular basis (minimum of four times daily) for a full work week (Monday through Friday). Fugitive air emission monitoring will then be conducted during soil excavation, handling and backfilling operations in accordance with the Health and Safety Plan.

TSP air sampling stations will be established around the perimeter of the Removal Action area consisting of a minimum of one upgradient and two downgradient locations. Locations will be chosen based on local wind data, so as to provide for upwind and downwind concentrations of dust and lead. Proposed locations are depicted on Figure FSAP-1.

Every attempt will be made to maintain the following siting recommendations regarding location of the high-volume samplers:

a. Sampler should be at least 60 feet from trees, buildings, or other large obstacles. A general placement rule is that the sampler should be located at least twice as far away from the obstacle as the height of the obstacle.

b. Sampler inlet should be 6 to 21 feet above the ground surface.

c. Sampler must have unrestricted air flow.

d. Sampler inlet should be at least 6 feet from any other high-volume sampler inlet.

e. The sampler cannot be placed directly upon the ground.

f. The sampler cannot be placed near exhaust flues or vents.

Final TSP locations will be determined during site mobilization based upon site logistics (electrical source, accessibility, etc.) and prevalent wind directions.

4.1 HIGH VOLUME TSP AIR SAMPLING

TSP air samples will be collected to determine total suspended particulate concentrations in accordance with the SOW. The TSP samplers will be operated continuously over 24-hour periods, except for brief down time periods due to change out of filters or repositioning of the samplers.

The samplers will be assembled according to manufacturer's instructions and attached to a stand. The filter disks will be changed each day. Filters will be sent to Pace Analytical in Indianapolis, Indiana every fifth working day for total lead analysis as well as total mass collected. Filters sent in for laboratory analysis will be submitted with a request for 24-hour turnaround of analytical services. Conditions at the site will be maintained such that the action level of 187.5 $\mu g/m^3$ are not violated at the site perimeter. The action level was derived from one half of the National Ambient Air Quality Standards (NAAQS) for particulate matter of 150 $\mu g/m^3$ converted to a one hour averaging period (USEPA, 2001). Air samples will also be analyzed for total lead during the removal action so as to maintain the NAAQS for lead particulate of 1.5 $\mu g/m^3$ for a 24-hour average of lead (based on a quarterly average) throughout the duration of the removal action.

4.2 LOW VOLUME PERSONAL/AREA AIR MONITORING

Air quality samples will be collected to determine the amount of antimony, arsenic, cadmium, and lead in the air for worker safety. These samples will be collected with five (5) low volume sampling pumps and sample cassettes. The sampling pump will be positioned upon personnel or in or near areas of potential fugitive dust emissions generation. Low volume personal/area air monitoring is described in detail in Section 7.1.4 and 7.1.5 of the HASP. The SOP for the personal is presented in Attachment FSAP-3.

4.3 CORRECTIVE MEASURES

The air monitoring methodologies described above will dictate engineering controls to ensure worker safety and that no potential impacts occur to surrounding residential areas. Corrective measures relating to spills, emergency contacts and response operations are described in the Contingency Plan included with the HASP.

4.3.1 Fugitive Dust Emissions

Air dispersion of contaminated soil may occur from mechanical agitation of soil by earth moving or soil treatment equipment. Air quality around the workplace will be monitored throughout the project by stationary air monitoring devices located around the perimeter of the site to determine on site air contamination. The action level for lead concentration in the ambient air is 1.5 $\mu g/m^3$.

4.3.2 Prevention of Fugitive Dust Emissions

Adequate dust control measures will be implemented throughout the project. Personal/area and stationary sampling devices will provide actual airborne concentration data for lead and particulate

matter. If dust generation is observed, the operation will be suspended or modified until corrective measures are taken to reduce the fugitive dust emission. Corrective measures may include wetting the area of concern, application of a surfactant to the contaminated surfaces, and/or filtering or otherwise controlling contaminated air.

5.0 DATA QUALITY OBJECTIVES

5.1 DATA QUALITY NEEDS, DUPLICATES, AND BLANKS

A combination of two levels of data quality objectives will be utilized in this project to address field screening and laboratory analytical data. Data Quality Objective Level 1, field-screening methods will be used for the XRF screening activities. Data Quality Objective Level 4 samples will be used for samples analyzed in the laboratory for confirmation of the clean up criteria and treatment prior to placement on-site and consolidation. Samples will be analyzed for the total lead or TCLP lead as outlined in the QAPP.

Rinsate blanks and field duplicates will be collected at a ten percent frequency interval for field quality assurance (QA) and quality control (QC), as well as laboratory QA and QC to be performed for all samples submitted to the laboratory. The laboratory QA/QC includes one matrix spike and one matrix spike duplicate (MS/MSD) for every 20 samples. A complete description of all QA/QC procedures is presented in the Quality Assurance Project Plan (QAPP) (Appendix D of the RD/RA Workplan).

5.2 DETECTION LIMIT REQUIREMENTS

The level of concern for each parameter directly affects the data quality requirements. Therefore, the sampling and analysis methods must be accurate at the level of concern. Furthermore, it is necessary that the analytical technique chosen has a detection limit well below the level of concern. Analytical methods that can accurately quantify constituents below their levels of concern will be used for the MMI sample analyses. The detection limits will generally be much less than the levels of concern. It is necessary that data quality objectives be consistent with clean-up levels or other levels.

Analytical detection limits should be less than the level of concern for each constituent and will be selected so that any analyzed parameter result can be compared to the appropriate level. The QAPP discusses the planned detection limits for analyses along with the methods to be used for this investigation in order to address the various levels for comparison.

5.3 CHAIN-OF-CUSTODY PROCEDURES

Proper documentation of sample collection and the methods used to control these documents are referred to as Chain-of-Custody (COC) procedures. COC procedures are essential for presentation of sample analytical results as evidence in litigation or at administrative hearings conducted by regulatory agencies. COC procedures also serve to minimize loss or misidentification of samples and to ensure that unauthorized persons do not tamper with collected samples. Section 5 of the QAPP describes all COC procedures for both field use and laboratory use. An example COC record form is also presented in the QAPP (Appendix D of the RD/RA Workplan).

5.4 SAMPLE SHIPPING

For shipping, all samples will be packaged in such a manner as to prevent damage or breakage during shipment or transport. For backfill samples that include VOC or SVOC analyses, the samples will need to be stored on ice during collection and shipment. Samples not delivered to the laboratory will be shipped through an overnight parcel service by sampling personnel. Samples will be placed into suitable containers, labeled and sealed in such a manner that tampering with the seal would be obvious. All sample holding times will be tracked and a copy of the Chain-of-Custody form will accompany the samples in a sealed plastic bag. Sample shipping is discussed in Section 4.3 of the QAPP (Appendix D of the RD/RA Workplan).

6.0 FIELD INSTRUMENT MAINTENANCE AND CALIBRATION

6.1 X-RAY FLUORESCENCE ANALYZER

The Spectrace 9000 energy dispersive X-Ray Fluorescence analyzer will be the instrument utilized for screening total lead concentrations in soil. The Spectrace 9000 instrument utilizes three radioisotope sources. Each source emits a different energy (wavelength) of radiation, which provides efficient analysis of specific ranges of elements. A 60-second scan time will be utilized for the duration of the Removal Action. Only qualified analysts trained in the proper use, theory, and safety of XRF analysis will operate this system.

The principle of XRF analysis is based on electron excitation. Elemental atoms in a soil sample are irradiated with a beam of x-rays. Electrons in the atoms at lower lying energy levels are excited to higher energy levels. The vacancies left in the inner electron orbital make the atom unstable. Relaxation to the ground state occurs, resulting in the emission of x-rays characteristic of the excited elements. Thus, by examining the energies of the x-rays emitted by the irradiated soil sample, identification of elements present in the sample is possible. Comparing the intensities of the x-rays emitted from a given sample to those emitted from reference standards with known analyte concentrations allows quantification of the elements present in the samples. Prior to any on-site activities, the Spectrace 9000 will be properly calibrated in order to allow for accurate sample analysis. Calibration specific response factor/calibration study will be done to verify the concentrations of lead in soils as discussed in Section XI (A) of Attachment FSAP-1 (XRF SOP). During on-site activities, the XRF will be standardized daily utilizing referenced standards for quality assurance and quality control.

6.2 Air Sampling/Monitoring Equipment

TSP air sampler

The TSP air sampler collects air samples using a high-volume vacuum pump to pull air through a filter, depositing airborne agents on the filter. A SOP for the TSP air sampler is presented in Appendix FSAP-2 and includes instrument calibration, sample collection, and routine preventive maintenance.

Personal/area low volume air sampler

The low volume air sampler collects air samples using a low-volume vacuum pump to pull air through a filter cassette, depositing airborne agents on the filter. An SOP for the area and personal low-volume sampler is presented in Appendix FSAP-3 and includes instrument calibration, sample collection, and routine preventive maintenance.

7.0 FIELD DOCUMENTATION

Logs of daily activities will be used to record sampling activities. Since there will be several different types of sampling activities going on (e.g., air, XRF, soil, treated material, backfill), possibly at the same time, there may be several log books. These books will be bound and have consecutively numbered pages. Entries in the field logbook will be made in ink and will include: the name of the author; date and time of entry; location of activity; sample collection or measurement methods; number of samples collected; sample identification numbers; field observation and comments; sampling depth increment for soils; field measurements; locations of photographs; and any deviations from the sampling plan. The field logbook will be stored in the document control center at the job site when it is not in use. Upon project completion, all logbooks will become part of the file records.

8.0 REFERENCES

Compliance Technologies, Inc. (CTI), January 17, 1991a, *Groundwater Analyses Report for Master Metals, Inc., Cleveland, Ohi.o*

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ENTACT, April 24, 1998a. *Phase I Final Report for Time-Critical Removal Action at the Master Metals Site, Cleveland, Ohio.* Prepared for the EPA Region 5 on behalf of the Master Metals PRP Group in Response to the April 17, 1997 Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA.

ENTACT, Inc. (ENTACT), November 23, 1998b. *Phase II Engineering Evaluation and Cost Analysis (EE/CA Report for the Master Metals Site, Cleveland, Ohio.* Prepared for the EPA Region 5 on behalf of the Master Metals PRP Group in Response to the April 17, 1997 Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA.

ENTACT, Inc. (ENTACT), February 6, 1998c. *Final Report for Removal Activities at the Holmden Avenue Site, Cleveland, Ohio.* Prepared for the EPA Region 5 on behalf of the Holmden Avenue PRP Respondent Group .

ENTACT, Inc., May 9, 1997, *Phase I Time-Critical Removal Action Workplan for the Master Metals Site, Cleveland, Ohio.* Prepared for the EPA Region 5 on behalf of the Master Metals PRP Group in Response to the April 17, 1997 Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA.

United States Environmental Protection Agency (USEPA), _____,2001a. *Administrative Order by Consent Pursuant to Section 106 of the Comprehensive Environmental Response, Compensation and Liability Act of 1980 –Master Metal Superfund Site, Cleveland, Ohio*, Docket No. _____.

USEPA, _____, 2001b,. *Statement of Work (SOW) for the Design/Construction and Cleanup at the Master Metals Superfund Site, Cleveland, Cuyahoga County, Ohio.*

USEPA, March, 1999, U.S.EPA *Proposes Clean-up Plan for Master Metals Site, Cleveland, Ohio.* Office of Public Affairs, Region 5, Chicago, Illinois.

USEPA, April 17, 1997, *Administrative Order by Consent Pursuant to Section 106 of CERCLA issued by the USEPA. Docket No: V-W-97-C-402.*

TABLE FSAP-1: LIST OF PARAMETERS AND TEST METHODS BY TASK, FIELD SAMPLING AND ANALYSIS PLAN, Master Metal Inc. Site, Cleveland, Ohio

Test Description	Test Method	Extraction Method	Matrix	Frequency	Container	Preservation	Sample Size	Maximum Holding Time
Pre-Excavation and Post-Excavation Confirmation								
Total Lead	SW-6010B[1]	SW-3050	Soil	One (1) composite confirmation sample per grid cell if historic slag is not encountered	P/G[2]	None	100 g	6 months
Total Lead	XRF[3]	na	Soil	Four locations per grid for screening purposes only	Field Test	na[4]	na	na
Soil Treatment Confirmation								
TCLP Lead[5]	SW-6010B	SW-1311	Soil	1 per 250 cubic yards for first 1,000 cubic yards, then at intervals of 500 cubic yards thereafter	P/G	None	250 g	6 months
Air Monitoring								
Total Suspended Particulate (TSP)	40 CFR Part 50, App. B	40 CFR Part 50, App. B	Air	daily [6] (4 per Perimeter)	Filter	na	na	na
Total Suspended Particulate (TSP) for Lead	40 CFR Part 50, App. G	40 CFR Part 50, App. G	Air	daily [6] (4 per Perimeter)	Filter	na	na	na
Total Lead by low-vol Sampler	NIOSH 7300 7105 7082	NIOSH 7300 7105 7082	Air	daily	Filter Cassette	na	na	na
Random Air Monitoring (RAM)	Instrument Specific	Instrument Specific	Air	daily	Real-Time	na	na	na

TABLE FSAP-1 (Continued): LIST OF PARAMETERS AND TEST METHODS BY TASK, FIELD SAMPLING AND ANALYSIS PLAN, MASTER METALS, INC., CLEVELAND, OHIO

Test Description	Test Method	Extraction Method	Matrix	Frequency	Container	Preservation	Sample Size	Maximum Holding Time
Imported Backfill Material (3)								
Total Arsenic	SW-6010(1)	SW-3050	Soil	Initial, 1 four-part composite per source	P/G(2)	None	25 g	6 months
Total Barium	SW-6010	SW-3050	Soil	same as above	P/G(2)	None	25 g	6 months
Total Cadmium	SW-6010	SW-3050	Soil	same as above	P/G	None	25 g	6 months
Total Chromium	SW-6010	SW-3050	Soil	same as above	P/G	None	25 g	6 months
Total Lead	SW-6010	SW-3050	Soil	same as above	P/G	None	25 g	6 months
Total Mercury	SW-7471	SW-3050	Soil	same as above	P/G	None	25 g	28 days
Total Selenium	SW-6010	SW-3050	Soil	same as above	P/G	None	25 g	6 months
Total Silver	SW-6010	SW-3050	Soil	same as above	P/G	None	25 g	6 months
Volatile Organic Compounds (VOCs)	SW-8260A	SW-5030	Soil	Initial, one grab sample per source	G	Cool, 4C	250 g	14 days
Total Petroleum Hydrocarbons (TPH)	SW-8015 Modified DRO	SW-3054, 3055, 5030	Soil	Initial, 1 grab sample per source	G	Cool, 4C	250 g	ext.-14 days anal.-40 days
Pesticides & PCBs	SW-8081	SW-3540/50	Soil	Initial, 1 four-part composite per source	P/G	Cool, 4°C	250 g	14 days
Total Lead	SW-6010	SW-3050	Soil	Four-part composite each 10,000 cubic yards	P/G	None	25 g	6 months

Notes:

(1) Sample Test Method designated as SW-xxxx is from EPA SW-846.

(2) P/G - Plastic or Glass

(3) XRF - X-Ray Fluorescence Analyzer (6) Analyze 1 every 5 days and, if RAM indicates a daily average exceeding 0.15 mg/m^3.

4) na - not applicable

(5) TCLP - Toxicity Characteristic Leaching Procedure

MASTER METALS SITE

Cleveland, Ohio

Figure FSAP-1

HISTORICAL SOIL SAMPLING LOCATIONS

ENTACT October 1, 1997

Description	Sample I.D.	Boring Depth	Total Pb Result (ppm) & Sample Depth (Ft.)
Slag	B-1	Refusal @ 4'	23 (2-3')
Slag	B-2	Refusal @ 4.5'	28 (2-3')
Slag	B-3	Refusal @ 5'	38 (2-3')
Slag	B-4	Refusal @ 5'	36 (2-3')
Brown Sand/Brick	B-5	6" Concrete 10' Sand/Fill	17 (2-3') 18 (8-10')
Slag	B-6	8" Concrete - Refusal @ 4.5'	40 (2-3') 32 (4-5')
Slag	B-7	6" Concrete Refusal @ 5'	2,625 (3')
Slag	B-8	4" Concrete Refusal @ 6'	1,400 (3')
Slag	B-9	4" Concrete Refusal @ 5'	3,825 (3')
Sand/Brick	B-10	12'	970 (3-5') 11,825 (8-10')
Sand/Silty Clay	B-11	10'	11,175 (3-5') 3,500 (8-10')
Sand/Clay	B-12	10'	52 (3-5') 1,200 (8-10')
Sand	B-13	6" Concrete 10'	975 (3-5') 650 (8-10')
Sand/Silty Clay	B-14	6" Concrete 10'	125 (3-5') 105 (8-10')
Sand	B-15	8" Concrete 10'	500 (3-5') 166 (8-10')
Sand/Silty Clay	B-16	10'	15 (3-5') 8 (8-10')
Sand/Silty Clay	B-17	10'	18 (3-5') 33 (8-10')
Sand/Silty Clay	B-18	10'	22 (3-5') 15 (8-10')
Sand/Silty Clay	B-19	10'	128 (3-5') 63 (8-10')
Slag	B-20	Refusal @ 5'	55 (4')
Slag	B-21	4" Concrete 10'	102 (8-10')
Slag	B-22	4" Concrete Refusal @ 5'	352 (3-5')
Slag	B-23	4" Concrete Refusal @ 1.5'	No Information Available
Slag	B-24	6" Concrete Refusal @ 2.5'	4,960 (2')
Sand/Silty Clay	B-25	6" Concrete 10'	5,010 (3-5') 650 (8-10')
Slag	B-26	8" Concrete Refusal @ 7'	1,120 (3-5')
Sand	B-27	6" Concrete Refusal @ 1.5'	14,070 (1')
Slag	B-28	6" Concrete Refusal @ 5'	1,300 (4-5')
Slag/Sand	B-29	6" Concrete Refusal @ 5'	225 (3-5')
Slag/Coal	B-30	6" Concrete 10'	1,260 (3-5') 32 (8-10')
Slag	B-31	Refusal @ 5'	229 (5')
Trench Drain Sediment	SS1	Near Surface	115,000 + TCLP 1,230
Trench Drain Sediment	SS2	Near Surface	8,610 + TCLP 1,040
Surface Soil	SS3	Near Surface	98,000 + TCLP 1,220
Surface Soil	SS4	Near Surface	6,020 + TCLP 3.3
Low Area Sediment	SS5	Near Surface	78,340 + TCLP 959
Surface Soil	SS6	Near Surface	94,000 + TCLP 1,060
Surface Soil	SS7	Near Surface	107,000 + TCLP 1,260
Surface Soil	SS8	Near Surface	24,000 + TCLP 6.3
Surface Soil	SS9	Near Surface	24,200 + TCLP 6.3
Surface Soil	SS10	Near Surface	43,100 + TCLP 757

NOTES:

- Site Plan Not To Scale
- Site Features And Boring Locations are Approximate

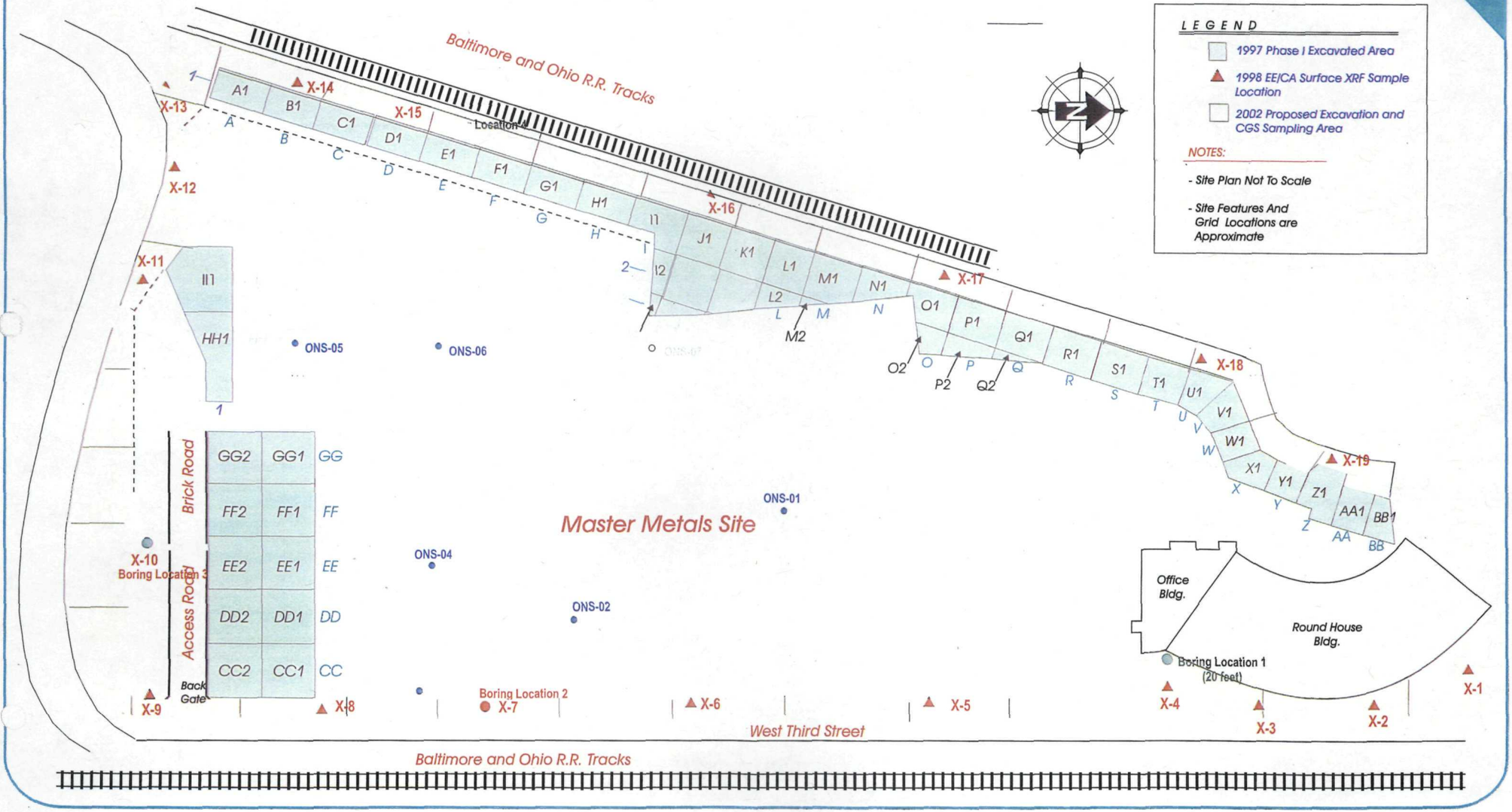
LEGEND

- Boring Location - CTI; 1990
- Boring Location/Monitoring Well - CTI; 1990
- Surface Grab Sample Location- E & E; 1992
- Fence
- RailRoad Tracks

MASTER METALS SITE
Cleveland, Ohio

ENTACT

Figure FSAP-2
PROPOSED EXCAVATION AND COORDINATE GRID SYSTEM SAMPLING AREAS



ATTACHMENT FSAP-1

XRF Standard Operating Procedure

X-RAY FLUORESCENCE (XRF) ANALYSIS OF SOIL STANDARD OPERATING PROCEDURES

FOR

THE MASTER METALS, INC. SITE CLEVELAND, OHIO

PREPARED BY:
ENTACT & Associates, LLC.

March, 2001

Table of Contents

I. Principle, Scope and Application

The purpose of this standard operating procedure (SOP) is to serve as a guide for the field analysis of soils for metals. The procedures herein are general operating procedures for the Spectrace 9000 XRF Analyzer or equivalent. They contain detailed procedures for calibration, operation and maintenance of the XRF.

X-radiation of sufficient energy will cause all atoms to fluoresce, emitting x-rays of characteristic energy. By analyzing the fluorescent radiation emitted by a sample under excitation, both the identity and the quantity of the elements present in the sample can be determined.

II. Parameters To Be Measured

A. Lead is the contaminant of concern at this site and will be the only metal measured and reported by the XRF.

III. Range Of Measurement

A. The range of measurements for lead is 50 ppm through 300,000 ppm.

IV. Detection Limit

A. The detection limit is variable with each analysis. The detection limit for each analysis is three times the XRF calculated standard deviation.

Example:
1. The XRF calculated standard deviation is 5 ppm.
2. 5 X 3 = 15
3. The detection limit is 15 ppm.

V. Sample Matrix

A. The SOP is applicable to both in-situ and ex-situ soils and waste.

VI. Interferences and Corrective Actions

A. Lead - Arsenic Interference

1. Interference

Due to the close proximity of the spectra for lead and arsenic, arsenic levels may be masked when the arsenic levels are less than 10% that of lead.

2. Corrective Action

TN Technologies has developed an additional software package for the Spectrace 9000 that will allow the XRF to detect arsenic when levels are as low as 5% that of lead.

B. Moisture

1. Interference
   High moisture content (approximately 20% moisture) of muds and sludges can cause erroneous results.

2. Corrective Action
   Soils containing high moisture content should be dried prior to analysis.

C. Matrix Effects

1. Interference
   Physical characteristics such as particle size and homogeneity can affect the accuracy of the analysis.

2. Corrective Action
   Whenever a new matrix is encountered a sample should be analyzed by both XRF and the laboratory analysis to ensure the XRF accurately analyzes the constituents in the matrix.

D. Placement

1. Interference
   If the XRF probe is not placed on a flat uniform soil location errors can result from the distance between the probe and the soil.

2. Corrective Action
   Ensure with each measurement that the probe window is placed flat against a uniform flat surface.

VII. Safety Precautions and Emergency Procedures

The State of Ohio Department of Health, Bureau of Radiation Protection will be properly notified prior to bringing the XRF instrument to the site.

A. Radiation Levels

According to the Spectrace 9000 users manual, the radiation exposure rate due to the XRF sources with the shutters closed is <0.1 mR/h. In addition, while the shutters are open, the exposure ate remains low provided a sample is completely covering the probe window. The XRF should never be run without a sample over the probe window.

B. Shipment

Under U.S. DOT regulations (49 CFR, 173.422) and International Air Transport Association (IATA), the XRF unit is classified as "Radioactive material, excepted

package, instruments, UN2910." As such, the device can be transported by any mode- air, land or sea. It is eligible to be transported in the baggage compartment of a passenger-carrying aircraft. The device is excepted from all specification packaging, marking and labeling. The bill of lading should, however, contain the words: "Radioactive material, excepted package, instruments, UN2910."

C. Storage

ENTACT's XRF units are licensed and permanently stored in the ENTACT Wood Dale office. The units can be transported to and temporarily (less than 30 days) stored in another state without the state being notified. If it is going to be transported to and stored in another state for longer than 30 days, that state must be contacted to determine the process involved with registering the XRF in that state.

D. Emergency Procedures

1. Secure the area around the incident. Keep unauthorized persons away. Alert people in vicinity of radioactive material and possible hazards.

2. DO NOT LEAVE THE SITE. Send a helper to notify the following persons:

   Radiation Safety Officer (RSO): Pat Vojack
   Work Phone: (630) 616-2100 Cell Phone: (630) 842-9860
   Home Phone: (847) 698-7508
   and
   Local Fire and Police Departments 911

3. The Radiation Safety Officer will provide appropriate notification to:

   Ohio Department of Health, Bureau of Radiation Protection: (614)644-2727
   and
   TN Technologies Inc.: (512) 388-9285 or (512) 388-9287

4. The RSO or alternate should inform emergency workers of the potential for existence of a radiation hazard; should help keep the area secure; and should explain to emergency personnel the location of the radioactive device and the extent of the possible hazard. In no case should the response personnel leave the site until qualified experts arrive, unless the worker is seriously injured or incapacitated, and must be removed from the site by emergency personnel.

   If the RSO cannot be reached, notify Don Self.

   Work Number: (972) 580-1323
   Home Number: (972) 475-2737

VIII. Sample Size, Collection, Preservation and Handling

A. The sample size, collection and handling requirements for samples undergoing XRF analysis are determined on a site specific basis. These are to be addressed in the site work plan and quality control plan. The exact requirements will vary depending on the use of the XRF on the site. No preservation is required for soils that are to be analyzed for metals.

IX. Apparatus and Materials

A. Probe

The probe consists of a sealed aluminum enclosure containing a high resolution mercuric iodide detector and three radioisotope x-ray excitation sources. The probe aperture window, through which the analysis is performed, is sealed with thin replaceable film. The probe also contains a pre-amplifier and bias supply for the detector and a mechanism to move the radioisotope sources from their shielded location during an analysis.

B. Electronics Unit

The electronics unit provides data acquisition, processing, and display capabilities. The computer includes a math coprocessor for fast calculation of results. Sufficient memory is available to store up to 300 sets of analysis and 120 spectra. An RS-232 port allows stored data to be transferred to another computer. The graphics display allows direct viewing and qualitative analysis of the x-ray spectra. The replaceable and rechargeable internal battery provides for field portable operation.

C. Additional Parts and Accessories

Additional parts and accessories include: the interconnecting able, battery chargers, RS-232C interface cable, carrying case, carrying bag, spare battery, analysis stand, Teflon bank and metal standards.

X. Routine Preventative Maintenance

-ENTACT identifies each XRF result with a unique identification number which all routine preventative maintenance to be accomplished as follows:

A. Standardization

The XRF must be standardized by technicians at TN Technologies on an annual basis.

B. Leak Tests

The XRF must be leak tested by technicians at TN Technologies every six months.

C. Source Change

The sources on the XRF must be changed by technicians at TN Technologies according to the following schedule:

Cd-109	2.5 years
Fe-55	5 years
Am 241	never

D. Film change

The film covering the aperture window needs to be changed whenever it is damaged.

XI. Calibration Standards

A. Site Specific Standards

1. Preparation

a. Collect three soil samples from the site in which XRF analysis will be performed. Use the XRF to guide the collection process. Attempt to collect samples that vary over the range of total lead levels detected at the site during the EECA (refer to Figure 1-3 of RD/RA Workplan).

b. Transport the samples to the lab and instruct the analyst to perform the following in the order listed for each sample:

-Dry the samples
-Grind the samples into a fine powder, removing any rocks or debris
-Homogenize each individual sample
-Split each sample. Return one half to ENTACT for use as the standard. Analyze the other half five times for total lead. The lab should then average the results giving a "certified value".

c. Prepare the site-specific standards using the returned portions of the samples. Place the soil into the XRF sample cups, cover with film and seal. The total lead value of the standard is the average of the five laboratory total lead values.

d. Use three of the prepared standards to check the standard daily for a calibration check

2. Storage

a. The standards must be stored in a manner that will prevent damage to the film.

b. The shelf life of the site-specific standards is 6 months. Upon expiration of these standards, the standard value should be re-certified by submitting additional sample to the laboratory for re-

analysis.

B. Teflon

1. Storage

a. The Teflon standard must be stored in a manner that will prevent damage and contamination.

b. These standards have an unlimited shelf life.

C. Pure Metal Standards

1. Storage

a. The five pure metal standards (lead, iron, tin, titanium and zinc) standards must be stored in a manner that will prevent damage and contamination.

b. These standards have an unlimited shelf life.

XII. Calibration Procedures

-The following procedures should be performed at the beginning of each days analysis. In addition one site-specific standard to be analyzed for every twenty sample locations analyzed. Finally, at the end of the day all three site-specific standards should be re-analyzed.

A. Instrument Set-up

1. Place the electronics portion of the XRF on a flat surface, adjusting the handle to be used as a stand.

2. Connect the Electronics portion to the probe using the interconnecting cable.

a. When inserting the cable into the probe and electronics portion, pull back metal cover on end of the cable, align the red dot on the cable with the grove on the insertion point and finally gently insert the cable until you hear a soft "click".

3. Remove the safety cover from the probe.

4. Place the probe on the lab stand base.

5. Secure the shield cup to the top of the probe.

B. Turn on procedures

1. Turn on the unit.

a. Press the "On" button.

b. You will then receive the prompt, "Is 0:00:00 the correct time?". If it is the correct time, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the time.

c. You will then receive the prompt, "Is 0:00:00 the correct date?". If it is,the correct date, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the date.

d. Allow the XRF to warm-up for at least 10 minutes.

C. Calibration

1. You are now at the main menu. Select measure (press the number 1 button).

2. You now need to modify the scanning time to allow 50 seconds per source to scan the iron standard.

   a. Select "modify" (press the number 1 button).

   b. Select the "Mod" (press the number 3 button).

   c. Enter 50 and press the Cont/Pause button.

   d. Select "Down" (press the number 2 button).

   e. Select "Mod" (press the number 3 button).

   f. Enter 50 and press the Cont/Pause button).

   g. Select "Down" (press the number 2 button).

   h. Select "Mod" (press the number 3 button).

   i. Enter 50 and press the Cont/Pause button.

   j. Select "Exit" (press the number 6 button).

3. You are now ready to analyze the iron (FE) standard.

   a. Place the iron standard over the source window.

   b. Close the shield cup lid.

c. Press the Cont/Pause button.

d. You will now see the label screen.

e. Select the column with "F" in it (press the number 2 button).

f. Select "F" (press the number 6 button).

g. Select the column with "E" in it (press the number 2 button).

h. Select "E" (press the number 5 button).

i. Press the Cont/Pause button.

j. Select "Opts" (press the number 5 button).

k. Select "See raw data" (press the number 5 button).

l. Select "Cd109 33" (press the number 1 button).

m. Select "Intensities" (press the number 6 button).

n. Select "Down" (press the number 2 button) until you can read the value for iron (FE). This value should be between 0.98 and 1.02. If it is not, perform an energy calibration. The procedures for an energy calibration are discussed in Section D of this section.

o. Select "Quit" (press the number 6 button).

p. Select "Quit" (press the number 7 button).

q. Select "EXIT" (press the number 0 button).

4. You now need to modify the scanning time for all three sources to measure the Teflon standard.

a. Select "Measure" (press the number 1 button).

b. Select "modify" (press the number 1 button).

c. Select "Mod" (press the number 3 button).

d. Enter 200 and press the Cont/Pause button.

e. Select "Down" (press the number 2 button).

f. Select "Mod" (press the number 3 button).

g. Enter 200 and press the Cont/Pause button.

h. Select "Down" (press the number 2 button).

i. Select "Mod" (press the number 3 button).

j. Enter 200 and press the Cont/Pause button.

k. Select "Exit" (press the number 6 button).

5. You are now ready to analyze the Teflon standard.

a. Place the Teflon standard over the source window.

b. Close the shield cup lid.

c. Press the Cont/Pause button.

d. You will now see the label screen.

e. Select the column with "T" in it (press the number 5 button).

f. Select "T" (press the number 6 button).

g. Select the column with "E" in it (press the number 2 button).

h. Select "E" (press the number 5 button).

i. Select the column with "F" in it (press the number 2 button).

j. Select "F" (press the number 6 button).

k. Select the column with "L" in it (press the number 3 button).

l. Select "L" (press the number 6 button).

m. Select the column with "O" in it (press the number 4 button).

n. Select "O" (press the number 3 button).

o. Select the column with "N" in it (press the number 4 button).

p. Select "N" (press the number 2 button).

q. Press the Cont/Pause button.

r. Press the zero button.

s. Select "Page down" (press the number 2 button).

t. For all results, the result divided by the standard deviation should be less than five (5). If it is not, acquire new background data are discussed in Section E of this section.

6. You now need to modify the scanning times for site specific calibration.

a. Select "modify" (press the number 1 button).

b. Select "Mod" (press the number 3 button).

c. Enter 40 and press the Cont/Pause button.
d. Select "Down" (press the number 2 button).

e. Select "Mod" (press the number 3 button).

f. Enter 10 and press the Cont/Pause button.

g. Select "Down" (press the number 2 button).

h. Select "Mod" (press the number 3 button).

i. Enter 10 and press the Cont/Pause button.

j. Select "Exit" (press the number 6 button).

7. You are now ready to analyze the site specific standards.

a. Place one of the site specific standards over the source window.

b. Close the shield cup lid.

c. Press the Cont/Pause button.

d. You will now see the label screen.

e. Select the column with the first letter or number of your standard name (press the appropriate number button).

f. Continue this process for the entire standard label.

g. Press the Cont/Pause button.

h. Press the zero button.

i. Select "Page down" (press the number 2 button).

j. Note the value for lead (Pb) or whatever element for which you are analyzing the samples.

k. Repeat steps c-j for the standard two more times. Each standard should be analyzed in triplicate.

l. The average of the three values found for the standard should be within ± 20% of the known value of the standard. If it is now, perform an energy calibration. The procedures for an energy calibration are discussed in Section D of this section.

m. Repeat steps a-l for all other site specific standards.

The XRF is now ready to be used.

D. Energy Calibration

1. You are now at the Main menu. Select measure (press the number 1 button).
2. Select "Options" (press the number 5 button).
3. Select "Energy calibration" (press the number 1 button).
4. The XRF will then say "Measure Safety Cover".
5. Put the safety cover on the probe.
6. Select "Proceed" (press the number 1 button).
7. The XRF will return to the analysis screen when the energy calibration is complete.

E. Background Data Acquisition

1. You are now at the Main menu. Select measure (press the number 1 button).
2. Select "Options" (press the number 5 button).
3. Select "Acquire background data" (press the number 2 button).
4. The XRF will then say "Measure Quartz".

5. Put the quartz standard on the probe.
6. Select "Proceed" (press the number 1 button).

7. The XRF will return to the analysis screen complete and give further instructions. Follow these instructions until acquisition is complete.

XIII. Sample Preparation

A. In-situ Samples

1. Clear the soil of all vegetation.
2. Clear the soil of any debris that may puncture the aperture window.
3. Tamp the soil to ensure it is flat and free of voids.

B. Collected Samples

1. Dry the samples in an oven or microwave oven.
2. Grind the samples into a fine powder, removing any large rocks or debris.
3. Homogenize the sample to ensure consistency.
4. Place the soil into an XRF soil cup, cover with film and seal.

XIV. Analytical Measurement

A. Instrument Set-up

1. Place the electronics portion of the XRF on a flat surface, adjusting the handle to be used as a stand.

2. Connect the Electronics portion to the probe using the interconnecting cable.

a. When inserting the cable into the probe and electronics portion, pull back metal cover on end of the cable, align the red dot on the cable with the grove on the insertion point and finally gently insert the cable until you hear a soft "click".

3. Remove the safety cover from the probe.

B. Turn on procedures.

1. Turn on the unit.

a. Press the "On" button.

b. You will then receive the prompt, "Is 0:00:00 the correct time?". If it is the correct time, press "yes" (the number 1 button). If it is now the correct time, press "no" (the number 2 button). The XRF will then instruct you on how to reset the time.

c. You will then receive the prompt, "Is 0:00:00 the correct date?". If it

is the correct date, press "yes" (the number 1 button). If it is not the correct date, press "no" (the number 2 button). The XRF will then instruct you on how to reset the date.

d. Allow the XRF to warm-up for at least 10 minutes.

C. Field use

1. You are now at the main menu. Select measure (press the number 1 button).

2. You now may need to modify the scanning time.

   a. Select "modify" (press the number 1 button).

   b. Select "Mod" (press the number 3 button).

   c. Enter 40 and press the Cont/Pause button.

   d. Select "Down" (press the number 2 button).

   e. Select "Mod" (press the number 3 button).

   f. Enter 10 and press the Cont/Pause button.

   g. Select "Down" (press the number 2 button).

   h. Select "Mod" (press the number 3 button).

   i. Enter 10 and press the Cont/Pause button.

   j. Select "Exit" (press the number 6 button).

3. You are now ready for analysis.
   a. Place one of the sample over the source window or place the probe on the area to be analyzed making sure the window is not punctured.

   b. Close the shield cup lid if applicable.

   c. Press the Cont/Pause button.

   d. You will now see the label screen.

   e. Select the column with the first letter or number of your sample name (press the appropriate number button).

   f. Continue this process for the entire sample label.

g. Press the Cont/Pause button.

h. Press the zero button.

i. Select "Page down" (press the number 2 button).

j. Note the value for lead (Pb) or whatever element for which you are analyzing the samples.

k. Repeat steps c-j for the sample two more times. Each sample should be analyzed in triplicate.

XV. Data Treatment

A. The result at each sample location is recorded

B. All readings must be greater than three times the XRF calculated standard deviation in order to be considered valid.

Reading > 3 * Standard deviation

If the above level is not achieved increase the scan time until it is achieved.

XVI. Data Deliverables

-The following documents are available to the client upon request:

A. A summary of initial, ongoing and end of analysis calibration results. This should include each reading, the average of the three readings for each sit-specific standard and the percent difference between the result and the laboratory determined value.

B. A logbook detailing the following:

1. Weather conditions
2. Sampler/s
3. Date of analysis
4. Time of each analysis
5. Location of each analysis
6. Sample preparations required
7. Results of each analysis
8. Any problems encountered and corrective actions taken
9. List date of XRF purchase, latest calibration, leak test and source replacement

C. A printout of all results saved on the XRF and downloaded to a PC. This will be downloaded and formatted in EXCEL and will include sample number, date taken and value in ppm.

D. A summary of all QC required. This will be determined on a site specific basis.

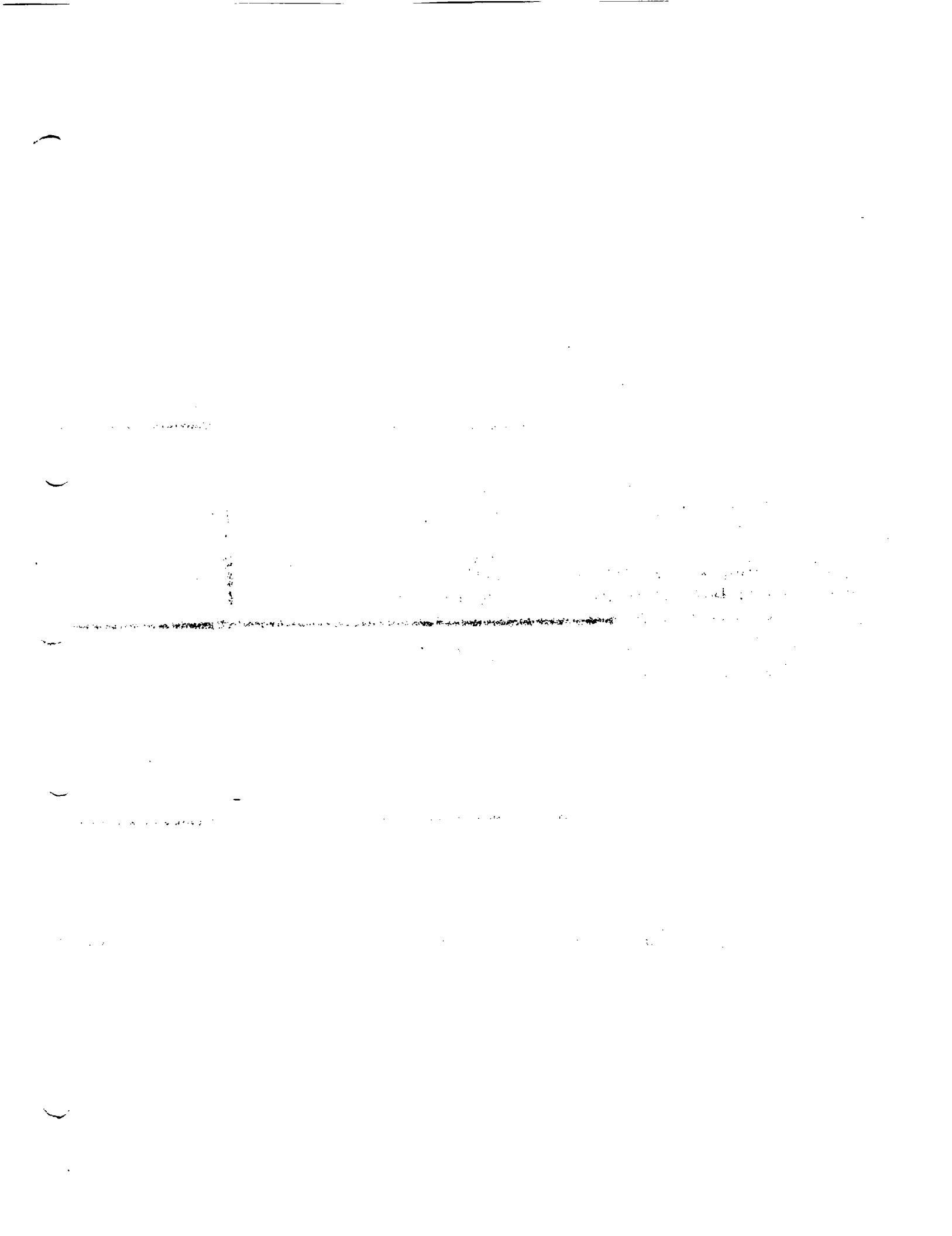
XVII. Quality Control Requirements

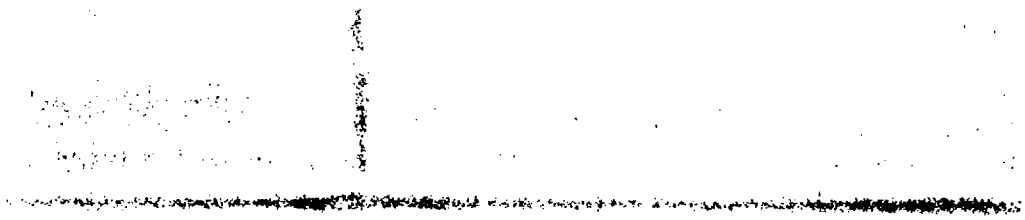
A. The quality control requirements for the use of the XRF are determined on a site specific basis. These are to be addressed in the site work plan and quality control plan. The exact requirements will vary depending on the use of the XRF on the site. However, all plans should require instrument calibration prior to and after XRF usage.

XVIII. References

A. Spectrace 9000 Analyzer Manual
TN Technologies Inc.
1992, 1993 and 1994

B. Quality Assurance Technical Information Bulletin
US Environmental Protection Agency
Vol. 1, No. 4
May 1991





ATTACHMENT FSAP-2

TSP Standard Operating Procedure

TOTAL SUSPENDED PARTICULATE AIR SAMPLING STANDARD OPERATING PROCEDURES

FOR

THE MASTER METALS, INC. SITE CLEVELAND, OHIO

PREPARED BY:
ENTACT & Associates, LLC.

March 2002

TSP Air Sampling for Lead
Standard Operating Procedures
Table of Contents

I. Principle, Scope and Application

The purpose of this standard operating procedure (SOP) is to serve as a guide for the field collection and handling TSP high volume samples. The procedures herein are general operating procedures for the Graseby Mass Flow Controlled Total Particulate Sampling System or equivalent. They contain detailed procedures for calibration, operation and maintenance of these pumps.

Sample collection involves the use of a high volume vacuum pump to pull air through a filter, depositing airborne agents on the filter. The filter is then analyzed in an accredited laboratory to determine how much of the agent of interest was deposited on the filter. Then, using the volume of air collected, the airborne concentration of the contaminate can be determined.

II. Parameters To Be Measured

A. Airborne lead.

III. Range Of Measurement

A. Airborne lead.

1. 0.07 micrograms per cubic meter of air

* assuming 2,400 cubic meters of air collected

IV. Detection Limit

A. Airborne lead.

1. 0.07 micrograms per cubic meter of air

* assuming 2,400 cubic meters of air collected

V. Sample Matrix

A. This SOP is applicable to airborne contaminants.

VI. Sample Media

A. Glass filter.

VII. Interferences and Corrective Actions

A. Light scattering

High concentrations of dissolved solids can produce scattering during atomic absorption analysis. This can be corrected instrumentally.

B. The combination of sample collection and analysis standard deviations is on average seven (7) to nine (9) percent.

VIII. Safety Precautions and Emergency Procedures

A. Explosion

1. Do not operate the pump or change its battery pack in oxygen enriched atmospheres of in atmospheres containing combustible gases, vapors or other explosive materials. An explosion may occur.

IX. Apparatus and Materials

A. Pump assembly.

1. Lid assembly.
2. Blower / Motor.
3. Mass Flow Controller.
4. Timer.
5. Flow Recorder.
6. Filter Paper Cartridge.

B. Calibration System

1. Vari-Flo Orifice Unit.
2. Calibration air hose.
3. Top Loading Adapter.
4. Digital Manometer.
5. Carrying Case.

X. Routine Preventative Maintenance

1. Routine Inspections
   - Power cords
   - Filter Screen
   - Frame Gasket
   - Recorder Pen
   - Motor Tubing
   - Motor Flange Gasket and Cushion

2. Motor Brushes
   - Motor Brushes should be changed every 400-500 hours in operation.

3. Calibration System Calibration
   - The calibration system should be calibrated by the manufacturer on an annual basis.

XI. Calibration System

A. The Graseby Vari-Flo Gilibrator Calibration System is to be used for calibration operations.

XII. Calibration Procedures

A. Frequency

Samplers should be calibrated per manufacturers recommendations or as indicated in the workplan, quality control plan, order or permit.

B. Procedures

1. Assemble calibration equipment.

2. Install the vari-flow orifice.

3. Perform a leak check.

4. Record the date, time temperature, barometric, pressure, sampler unit number, person performing calibration and the serial number of the calibration orifice.

5. Turn on the unit and allow to warm up.

6. Adjust the orifice so that the manometer is reading approximately 1.0 inches of water.

7. Record the exact manometer reading as well as recorder chart reading.

8. Repeat step 6 & 7 for 2, 3, 4 and 5 inches of water.

9. Turn off the unit.

10. Using the computer air monitoring calibration calculation software, calculate the orifice flow rate (Qa), corrected recorder response (IC), the set flow rate (SFR) and set point recorder response (SSP).

11. Turn on sampler unit, allow to warm up and set to the SSP.

XIII. Sample Collection

A. Frequency

Samples are to be collected on a twenty-four (24) hour cycle as per the workplan, order or permit requirements.

B. Procedures

1. Remove the filter from its envelope. On the envelope, record the date and the sampling unit where the filter will be used.

2. Position the new filter in the appropriate filter paper cartridge.

3. Go to the appropriate air monitoring station.

4. Turn off the unit.

5. Record the time that the unit was turned off on the used recorder chart.

6. Remove and cover the used filter paper cartridge.

7. Position the new filter paper cartridge and secure to the unit.

8. Close the collection unit hood.

9. On a new recorder chart, record the current date, time and sampler unit number.

10. Turn on the unit.

11. Check to see that the unit is operating at the correct recorder response point.

12. Close the unit door.

13. Return the filter paper cartridge and recorder chart to the site office / lab facilities.

14. Remove the filter from the filter paper cartridge.

15. Fold the filter in half, in on itself and place back into its original envelope.

16. Seal the envelope.

17. Using the collection time and sampler air flow rate, calculate the total volume of air collected.

18. Fill out the daily air monitoring log using the calculation equations presented in Section XIV

19. As necessary, fill out chain-of-custody forms, label the filter envelopes and deliver the filter to the laboratory for analysis.

20. Save all unanalyzed filters and recorder charts for six (6) month or as otherwise noted in the workplan, order or permit.

21. Clean and properly store the filter paper cartridge for future use.

XIV. Calculations

A. Volume of air.

1. Flow rate (FR) is to be determined by looking up the average daily recorder chart reading on the calibration correlation table, generated by the computer air monitoring calibration calculation software, and reading the corresponding flow rate.

2. Use the flow rate and collection time in minutes to calculate the volume collected.

Volume = FR x time

ex. Volume = 2.0 L/min. x 640 min.

= 1280 L of air collected

XV. Analytical Measurement

- The analytical method cited in either the work plan or quality control plan should be used to analyzed the air samples. The method should be consistent with 40 CFR Part 50 Appendix G.

XVI. Quality Control Requirements

A. The quality control requirements for the use of the TSP units are determined on a site specific basis. These are to be addressed in the site work plan, quality control plan and the site health and safety plan. The exact requirements will be dependant on specific site and/or order requirements. However all plans should include the periodic analysis of filter lot blanks.

XVII. References

A. 40 CFR Part 50

B. Operations Manual for The Graseby Model GS2310 TSP Sampling System Mass Flow Controlled

C. Instruction and Operation Manual - High Volume PM10 Sampler

ATTACHMENT FSAP-3

Area/Personal Low-Volume Air Sampler
Standard Operating Procedure

PERSONAL & AREA LOW VOLUME AIR SAMPLING FOR LEAD STANDARD OPERATING PROCEDURES

FOR

THE MASTER METALS, INC. SITE CLEVELAND, OHIO

PREPARED BY:
ENTACT & Associates, Inc.

March, 2002

Personal & Area Low Volume Air Sampling for Lead
Standard Operating Procedures
Table of Contents

Personal & Area Low Volume Air Sampling for Lead
Standard Operating Procedures

I. Principle, Scope and Application

The purpose of this standard operating procedure (SOP) is to serve as a guide for the field collection and handling of personal / area air samples. The procedures herein are general operating procedures for MSA Escort pumps or equivalent. They contain detailed procedures for calibration, operation and maintenance of these pumps.

Sample collection involves the use of a low volume vacuum pump to pull air through a filter cassette, depositing airborne agents on the filter. The filter is then analyzed in an accredited laboratory to determine how much of the agent of interest was deposited on the filter. Then, using the volume of air collected, the airborne concentration of the contaminate can be determined.

II. Parameters To Be Measured

A. Airborne lead.

III. Range of Measurement

A. NIOSH Method 7082

0.05 mg/m^3 to > 1 mg/m^3 for a 200 L air sample.

B. NIOSH Method 7105

0.002 mg/m^3 for a 200 L air sample.

C. NIOSH Method 7300

0.005 mg/m^3 for a 500 L air sample.

IV. Detection Limit

A. NIOSH Method 7082

0.05 mg/m^3 for a 200 L air sample.

B. NIOSH Method 7105

0.002 mg/m^3 for a 200 L air sample.

C. NIOSH Method 7300

0.005 mg/m^3 for a 500 L air sample.

V. Sample Matrix

A. This SOP is applicable to airborne contaminants.

VI. Interferences and Corrective Actions

A. Avoid operating during periods of heavy rain or in areas in which water is being sprayed or misted. Filter damage may occur.

B. High concentrations of calcium, sulfate, carbonate, phosphate, iodide, fluoride or acetate can cause interferences during laboratory analysis. These can be offset by an additional sample treatment step.

C. Do not exceed a filter loading of 2 mg of total dust.

VII. Safety Precautions and Emergency Procedures

A. Explosion

1. Do not operate the pump or change its battery pack in oxygen enriched atmospheres or in atmospheres containing combustible gases, vapors or other explosive materials. An explosion may occur.

VIII. Apparatus and Materials

A. Pump assembly.

1. Pump.
2. Pump air hose.
3. Filter cassette.
4. Battery pack.

B. Calibration System

1. Calibration unit.
2. Calibration air hose.
3. Calibration fluid.

IX. Routine Preventative Maintenance

1. Battery Charge

- The battery should be charged following each days use to ensure proper operation.

2. Inlet filter check and replacement
   - The internal inlet filter should be checked periodically for particles and water and should be changed when clogged.

3. Calibration System Calibration
   - The calibration system should be calibrated by the manufacturer on an annual basis.

X. Calibration System

A. A Gilian Gilibrator Calibration System is to be used for calibration operations.

XI. Calibration Procedures

- The following procedures should be performed prior to and after each use of a personal / area pump.

A. Turn on the pump and calibration system. Allow the pump to operated in the environment to be sampled, with a filter cassette positioned as if actual sample was being collected for approximately 2 - 5 minutes prior to calibration.

B. Connect a filter cassette to the pump air hose. Connect the opposite cassette inlet to the calibration system.

C. Take five readings at the flow rate to be used during sampling with the calibration system. The average of the five is the initial flow rate.

D. At the end of the days operations, after the sample has been collected, repeat steps A - C. This will give the final flow rate. The flow rate is the average of the initial flow rate and the final flow rate.

XII. Sample Collection

A. Frequency

- Unless otherwise specified, personal air samples should be collected at the following frequency.

1. According to 29 CFR 1926.62, initial exposure determination should be made whenever there has been a change of equipment, process, control, personnel or a new task has been initiated that may result in additional employees being exposed

to lead at or above the action level of 30 $\mu g/m^3$ or the permissible exposure limit (PEL) of 50 $\mu g/m^3$, and as follows:

a. If the initial determination shows levels to be below of 30 $\mu g/m^3$, no further monitoring need be performed except as noted in XII (A) (1).

b. If the initial determination shows levels to be above 30 $\mu g/m^3$ but below of 50 $\mu g/m^3$, monitoring needs to be performed every 6 months or until two consecutive measurements, taken at least 7 days apart, show levels to be below 30 $\mu g/m^3$ at which time monitoring may be discontinued until there is a change in conditions as noted in XII (A) (I).

c. If the initial determination shows levels to be above 50 $\mu g/m^3$, monitoring needs to be performed quarterly or until two consecutive measurements, taken at least 7 days apart, show levels to be below 50 $\mu g/m^3$, at which time monitoring may be performed every 6 months. If two consecutive measurements, taken at least 7 days apart, show levels to be below 30 $\mu g/m^3$, monitoring may be discontinued until there is a change in conditions as noted in XII (A) (I).

B. Procedures

1. Following initial calibration, connect a new filter cassette to the pump air hose.

2. Secure the pump onto the person being monitored, positioning the filter cassette inlet within the persons breathing zone (between the chest and nose).

3. Remove the end-cap from the filter cassette.

4. Turn on the pump.

5. Document the time that the pump was turned on, the person being monitored and the task he/she is performing.

6. Allow the pump to operate throughout the day's activities.

7. The pump is to be checked periodically during sample collection by the person performing the sampling, to ensure the pump is operating properly. At the end of

the day's activities, turn off the pump.

8. Replace the end-cap on the filter cassette.
9. Document the time that the pump was turned off.

10. Remove the filter from the pump air hose and replace the other end-cap on the filter cassette.

11. Label the cassette.

12. Calibrate the air pump.

13. Fill out all appropriate chains of custody and other required forms, and Prepare the cassette(s) for shipment to the laboratory.

XIII. Calculations

A. Volume of air.

1. Calculate the flow rate (FR) in mL/min. of air.

FR = (initial flow rate + final flow rate) / 2

ex. FR = (1500 mL/min. + 2500 mL/min.) / 2
= 2000 mL/min.

2. Convert flow rate into L/min.

Flow rate (FR) in L/min. = FR in mL/min. / 1000

ex. FR in L/min. = 2000 mL/min. / 1000
= 2.0 L/min.

3. Use the flow rate and collection time in minutes to calculate the volume collected.

Volume = FR x time

ex. Volume = 2.0 L/min. x 640 min.
= 1280 L of air collected

XIV. Analytical Measurement

- The analytical method cited in either the work plan or quality control plan should be

used to analyze the air samples. If no method is sited, one of the below methods should be used depending on the volume of air collected.

A. NIOSH Method 7082
   1. 200 L - 1500 L of air for time weighted average (TWA) measurements.

B. NIOSH Method 7105
   1. 1 L - 1500 L of air for TWA measurements.

C. NIOSH Method 7300
   1. 50 L - 2000 L of air for TWA measurements.

XV. Quality Control Requirements

A. The quality control requirements for the use of the personal / area pumps are determined on a site specific basis. These are to be addressed in the site work plan, quality control plan and the site health and safety plan. The exact requirements will vary depending on the use of the personal / area pumps on the site. However, all plans should require instrument calibration prior to and after personal / area pump usage.

XVI. References

A. 29 CFR 1926.62

B. NIOSH Manual of Analytical Methods
   Fourth Addition
   August 15, 1994

C. MSA Escort Pump Users Manual

APPENDIX D

FINAL QUALITY ASSURANCE PROJECT PLAN

QUALITY ASSURANCE PROJECT PLAN
FOR THE
MASTER METALS, INC. REMOVAL ACTION
Cleveland, Cuyahoga County, Ohio

March 2002

Prepared by:

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Quality Assurance Project Plan, Revision 0
Removal Design / Removal Action for the
Master Metals, Inc. Site
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LIST OF ACRONYMS/ABBREVIATIONS

ARARs	Applicable or Relevant and Appropriate Requirements
AOC	Area of Contamination
ASTM	American Standards for Testing Materials
BNA	Base-Neutral-Acid Extractables (Semivolatile Organics)
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act (Superfund)
COC	Chain of Custody
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limits
CRQL	Contract Required Quantitation Limits
CRL	Central Regional Laboratory
DCF	Document Control Management
DQO	Data Quality Objective
EMSL	Environmental Monitoring and Support Laboratory
FSAP	Field Sampling and Analysis Plan
GMPP	Groundwater Monitoring Program Plan
ICP	Inductively Coupled Plasma
ICVS	Initial Calibration Verification Standard
MMI	Master Metals, Inc.
MS/MSD	Matrix Spike/ Matrix Spike Duplicate
µg/kg	Micrograms/kilograms
NIST	National Institute of Standard Technology
NPL	National Priorities List
NTU	Nephelometric Turbidity Units
OSC	On-Site Coordinator
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PCBs	Polychlorinated Biphenyls
PM_{10}	Particulate matter less than 10 microns
ppb	Parts Per Billion
ppm	Parts Per Million
QA/QC	Quality Assurance/ Quality Control
QAMP	Quality Assurance Management Plan
QAPP	Quality Assurance Project Plan
RPD	Relative Percent Differences
RAS	Routine Analytical Services
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/ Feasibility Study
RD/RA	Remedial Design/ Remedial Action
RPM	Remedial Project Manager
SARA	Superfund Amendments and Reauthorization Act
SAS	Special Analytical Services
SMC	Sample Management Coordinator

SOP	Standard Operating Procedure
SRM	Standard Reference Materials
SOW	Statement of Work
SW846	Test Methods for Evaluating Solid Waste 1986.
TAL	Target Analyze List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TPH	Total Petroleum Hydrocarbon
TSP	Total Suspended Particulate Matter
USEPA	United States Environmental Protection Agency
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
XRF	X-Ray Fluorescence

1.0 PROJECT DESCRIPTION

1.1 INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been developed by ENTACT & Associates, LLC (ENTACT) for the Master Metals, Inc. Site for use in conjunction with the Removal Design/Removal Action (RD/RA) Workplan and Health and Safety Plan. These are distinct documents that form the project operations plan intended to guide field personnel, contractors, and other involved parties in all aspects of field operations. This QAPP will provide QA procedures for activities during the removal action performed in accordance with the 2002 Administrative Order of Consent (AOC) for the Master Metals Superfund Site located in Cleveland, Cuyahoga County, Ohio.

A Phase I Time Critical Removal (TCR) for lead-impacted materials has already been conducted at the MMI site to remove contamination that posed an immediate risk to human health. Following the Phase I TCR, the Phase II EE/CA investigation was performed to delineate and evaluate the nature and extent of lead contamination remaining at the site to determine the appropriate non-time critical removal action (RA) needed to address existing site conditions. The removal action covered under this QAPP will address the remaining lead contamination in soils within the site and along the site perimeter to complete all necessary remedial action in accordance with the AOC.

The United States Environmental Protection Agency (USEPA) policy requires that all remedial activities be under the control of a centrally managed QA program. This requirement applies to all environmental monitoring activities supported by the EPA. Each contractor that generates data has full responsibility to implement minimum procedures to ensure that precision, accuracy, representativeness, completeness, and comparability of these data are known. To meet this objective, this site specific QAPP has been prepared detailing QA/QC procedures to ensure data generated during the remedial activities are accurate, precise, comparable and complete and therefore, representative of site conditions.

This QAPP will serve as a controlling mechanism during the performance of the sampling and analysis activities to detail procedures to ensure that technical data gathered during the construction phase of the interim measures are accurate, precise, complete, and representative of actual field conditions and meet minimum requirements of the design. All QA/QC procedures will be structured in accordance with applicable technical standards, EPA requirements, and regulations in general accordance with the USEPA Region 5 Model RCRA QAPP guidelines.

1.2 SITE/FACILITY DESCRIPTION

1.2.1 Location

The Master Metals Site (the "Site") encompasses approximately 4.3 acres in the "flats" area of downtown Cleveland, a heavily industrialized sector of the city. The Site includes the former Master Metals Inc. lead facility (the "Facility") located at 2850 West Third Street, Cleveland and stockpiled soils removed from the surrounding contaminated residential property at 1157, 1159 and 1167 Holmden Avenue (the "Holmden Properties") where lead-impacted material from Master Metals was deposited as fill (USEPA, 1999). Railroad tracks border the site on two sides and the LTV Steel facility lies to the east and south. The Cuyahoga River is located approximately 1,500 feet to the east and athletic field and playground are situated approximately 1,000 feet to the west. The nearest residential property to the former facility is

approximately 2,000 feet to the northwest. (USEPA, 1999).

1.2.2 Local Geology, Hydrology and Hydrogeology

The glacial and post-glacial surficial material in the vicinity of the MMI site consists of tills, lacustrine, and fluvial deposits. The glacial deposits are generally less than 40 feet thick in the site area and overlay a Devonian/Pennsylvanian-aged bedrock consisting of unconsolidated shale and sandstone (E&E, 1993).

Site investigations conducted at the site between 1990 and 1998 indicate that fill is present beneath the site to an approximate depth of four feet, with native soils of silty clay found at five feet (WWC, 1990). The water table is encountered at an approximate depth of 10 feet (WWC, 1990).

1.3 SITE/FACILITY HISTORY

1.3.1 General History

The facility was constructed in 1932 on slag fill by National Lead Industries, Inc. (NL) who owned and operated the facility as a secondary lead smelter, producing lead alloys from lead-bearing dross and scrap materials. NL also engaged in battery cracking operations at this facility. In 1979, the facility was purchased from NL Industries by MMI who continued to run secondary lead smelter operations.

As part of their operations, the Master Metals facility received lead-bearing materials classified and regulated under Resource Conservation and Recovery Act (RCRA) as D-008 hazardous waste from off-site sources (USEPA, 1999). This waste was converted into lead ingots using pot and rotary furnaces equipped with baghouses to collect particulate matter from the furnace that consisted predominantly of lead dust. The sludge that accumulated in the furnaces after smelting was classified as K069 waste hazardous waste. Finished lead ingots were stored in a roundhouse at the north end of the property prior to shipment off-site.

Based on background information, the by-products produced from smelting operations included furnace flux, slag, dross, baghouse fines and furnace sludge (USEPA, 1999). With the exception of slag, which was tested and disposed of off-site, most of the lead-bearing by-products were recycled back into the furnace. Cooling water used in the operations was diverted to the City of Cleveland's sewer system.

On November 19, 1980, Master Metals filed a "Part A permit" pursuant to the newly-regulated RCRA requirement, and obtained an "interim status" under RCRA to operate specific waste piles and treatment units, as well as container-based storage area for the hazardous lead-bearing materials. On January 11, 1982, Master Metals filed for Chapter 11 bankruptcy through the U.S. Bankruptcy Court for the Northern District of Ohio but subsequently went into reorganization and operations at the facility continued. Though Master Metals had submitted a Part B RCRA application sometime prior to November 8, 1985, on that date the facility lost interim status for the hazardous lead-bearing waste piles at the facility for failure to comply with financial requirements of 40 CFR Part 265, Subpart H.

Violations relating to poor operating practices are documented in various state and federal agency reports. On June 15, 1987, a complaint of violations of RCRA was filed by the United States seeking closure of the D008 and K069 waste piles. In response to this action, Master Metals and the U.S. entered a Stipulation to resolve these RCRA violations as well as financial responsibility

1.3.2 Past Regulatory and Data Collection Activities

Numerous investigations have been conducted by MMI at the facility between 1990 to 1998 to determine the nature and extent of constituents of concern related to former operations. These investigations are summarized in the following subsections.

1.3.2.1 Compliance Technologies, December 1990

Compliance Technologies, Inc. (CTI) conducted a Phase II environmental assessment of the MMI site from December 3 through December 11, 1990. The investigation included the advancement of 31 soil borings to a maximum depth of 10 feet, and the installation of four monitoring wells to a depth of 15 feet to evaluate subsurface and groundwater conditions beneath the MMI facility and determine the impact of prior slag disposal/landfill activities on these media (CTI, 1991b).

Forty-four subsurface soil samples were collected from 31 borings located in or near the MMI facility. The samples were collected from depths ranging between two to ten feet below ground surface (CTI, 1991b). The soil samples were submitted to BHM Analytical Laboratory, Chagrin Falls, Ohio and analyzed for eight RCRA metals, including arsenic, barium, cadmium, chromium, lead, mercury, selenium, silver. The analytical results showed on-site lead concentrations ranging from 18.1mg/Kg to 14, 070 mg/Kg, with lead levels one to two orders of magnitude above the other metals detected. Off-site concentrations of lead in subsurface samples ranged from 7.85 to 55 mg/Kg. Slightly elevated concentrations of chromium and cadmium were observed in only 17 of the 44 samples. Sample locations and the associated lead concentrations are shown in Figure FSAP 1.

Groundwater was reported to be present between three to ten feet across the relatively flat facility. Four groundwater samples were collected from the newly-installed monitoring wells on December 28, 1990 using hand bailers and were not filtered. Total lead concentrations ranged between 0.45 mg/L to 1.39 mg/L.

In addition to the soil samples, two samples were collected the brick and slag material and analyzed for the TCLP 8 RCRA Metals, reactive sulfide, total cyanide, pH and flash point to determine if these materials were hazardous by characteristic (CTI, 1991b). Lead was present in the slag material at 7,075 mg/Kg with leachable lead detected in the slag material at 16.1 mg/L.

1.3.2.2 Ecology & Environment, July 1992

On July 14, 1992, Ecology and Environment (on behalf of the U.S. EPA) collected seven surface samples on-site (SS1 - SS7) and three off-site surface soil samples from outside the fence to the east, south and west (SS8 – SS10) as part of a site assessment and hazard evaluation of the MMI facility. All soil samples were submitted to American Environmental Laboratories, Inc. of Bedford, Ohio for analysis of the eight RCRA metals.

Lead concentrations in the on-site surface soil samples ranged from 6,020 to 115,000 mg/Kg. Off-site surface soil samples collected outside the fence showed lead concentrations ranging between 24,000 to 43,100 mg/Kg (E&E, 1992). Sample locations and the associated lead levels are presented in Figure 1-2. Once again, lead values were 1-2 orders of magnitude higher than the seven other metals. Some results

exhibited minor arsenic, barium, cadmium, and chromium concentrations, relative to the co-located lead concentrations (E&E, 1992).

In July 1992, Ecology and Environment (on behalf of U.S. EPA) collected samples proximate to the facility property to determine if the facility contaminants were subject to airborne transport. Analysis of these samples (SS8 - SS10) for RCRA metals showed total lead levels of 24,000 - 43,100 ppm (see Figure 2-1).

1.3.2.3 Phase I Time Critical Removal

As part of the time-critical removal, all exposed on-site surface areas (e.g., not covered by concrete) were excavated to a maximum depth of two feet or until slag fill material (e.g., slag, cinders, etc.) were encountered, whichever came first. XRF information collected from the floor of the excavations exhibited lead concentrations up to 39,000 ppm in the remaining slag fill material. The TCR also included the demolition, decontamination and off-site transportation of former facility structures. The activities are summarized in Section 1.4.1 of the RD/RA Workplan.

1.3.2.4 Phase II Engineering Evaluation and Cost Assessment

The on-site soil sampling included the advancement of seven borings on-site. Results indicated that 5 of the 7 borings exceeded 1,500 mg/Kg lead at total depth. Historic slag was encountered at approximately three to four feet which is consistent with the information collected during the Phase I TCR (ENTACT, 1998b. The soil sampling locations are illustrated in Figure 1-3 of the RD/RA Workplan. The on-site sampling indicated that significant lead concentrations, up to 35,000 mg/Kg, remained in on-site soils to a depth of 3 to 4 feet. These areas were either covered with the existing concrete surface or had been excavated and backfilled with 2 feet of clean fill as part of the Phase I TCR. Therefore in areas where the concrete was competent and in uncovered areas that were excavated as part of the Phase I TCR, the potential for further entrainment of airborne lead had been mitigated and was no longer considered a concern (ENTACT, 1998b). However a potential for airborne lead releases did exist in areas where the concrete was compromised. These areas were recommended for repair to mitigate this airborne migration route (ENTACT, 1998b)

A perimeter surface soil survey was conducted adjacent to the fence line along the western, eastern and southern boundaries of the MMI facility property using an XRF instrument, at nineteen locations designated in Figure 1-3. Results of the perimeter lead survey showed lead levels ranging from 931 ppm to 36,587 ppm within the upper 12 to 24 inches of soils, decreasing rapidly with depth. The surficial elevated lead levels currently pose a potential ingestion or inhalation threat, and were recommended for further remedial action (ENTACT, 1998b).

Off-site sampling included the collection of nine off-site surface soil samples along Quigley Avenue. The results showed levels of the average lead concentrations to be below the Superfund residential soil screening level of 400 mg/Kg indicating potential airborne lead impacts from the former MMI facility are minimal. No further action was recommended (ENTACT, 1998b).

Groundwater sampling conducted in 1991 showed total lead concentrations ranging from 0.45 mg/L to 1.35 mg/L, total chromium concentrations ranging from 0.02 mg/L to 1.33 mg/L and lesser concentrations of arsenic and cadmium (CTI, 1991). Groundwater sampling of the three existing monitoring wells during the 1998 EE/CA investigation showed the presence of lead, arsenic, cadmium

and chromium at levels that have either remained at, or have declined from, the 1992 sampling results. Groundwater is not used as a source of drinking water within a four-mile radius of the site, with Lake Erie supplying the greater Cleveland area with its drinking water supply. Based on the low concentrations of metals in the groundwater and the lack of any potential downgradient receptors, the groundwater migration pathway was eliminated as a concern (ENTACT, 1998b).

The EE/CA assessment verified that lead was the predominant hazardous constituent of concern at the site, with lesser occurrences of arsenic. Removal action directed at lead exceedences would also address the co-located elevated levels of arsenic. Based on a streamlined risk evaluation, a risk-based remediation goal (RBRG) for lead of 1,000 mg/Kg was established for on-site and off-site perimeter soils (ENTACT, 1998b). Based on the EE/CA results this final removal action has been designed to address the remaining lead impacts associated with former facility operations.

1.3.3 Current Status

Based on the findings of the Phase II EE/CA, an AOC was entered into between the USEPA and the PRP Respondent Group in Spring 2002 to perform a non-critical removal action outlined in the Statement of Work (SOW) to address remaining lead impacts at the site that are associated with former facility operations. In accordance to the revised Statement of Work (SOW), the following tasks are to be completed as part of this AOC:

- Clear and grub areas requiring excavation of all trees and brush for disposal off-site.
- Demolish above-grade concrete and metal structures remaining on-site after the Phase I TCR demolition activities in accordance to the design specifications. Sized concrete construction debris will either be used as a sub-base material in areas to be covered with the asphalt cover or will be transported off-site disposal as construction debris. All wood, bricks or metal debris that are removed will be disposed of off-site as construction debris.
- Establish a coordinate grid system along the perimeter of the property outside the fence line and in on-property areas where excavation is required.
- Excavate off-property soils along the western, eastern and southern perimeter of the MMI facility, that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first. XRF screening technology will be used to guide the depth of the excavations during removal.
- Excavate designated on-property soils that are not under concrete or the proposed asphalt cover (including grids I1, J1 and K1 excavated during the Phase I TCR) that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first.
- Conduct confirmatory soil sampling from the excavation floor in grids where the excavation was terminated prior to reaching the historic slag fill material to confirm that all soils that are above the cleanup level have been excavated and removed.
- Backfill all excavated areas once verified to have met the RBRG or have reached historic slag fill, and grading to promote positive drainage in accordance with the design documents. Backfill for areas

not covered by asphalt or concrete will be filled with clean imported fill material that has been approved for use based on analytical results and is suitable to maintain vegetative growth.

- Stabilize excavated soils to meet the applicable LDRs for contaminated soils for lead, and any underlying hazardous constituent (UHC) during waste profiling, to render the material nonhazardous for either use as fill in low areas beneath the proposed asphalt cover or for off-site disposal at an approved Subtitle D facility.

- Conduct verification sampling of treated soils using TCLP lead analysis to verify the material has been rendered non-hazardous for lead prior to either placement in low areas beneath the proposed asphalt cover or for off-site disposal as nonhazardous waste.

- Off-site disposal of all treated soils not used to fill low areas beneath the proposed asphalt cover, including stockpiled soils from the Holmden Properties Removal Action, in accordance with the SOW and the approved design plan.

- Place an asphalt cover over the deteriorated area of the concrete located in southern portion of the site in accordance with the design documents.

- Recondition existing concrete surfaces not under the asphalt cover by sealing any significant cracks and breaks that extend through the concrete surface, followed by encapsulation of the concrete surface, in accordance with the approved design plan.

- Abandon of all existing monitoring wells on site in accordance to applicable State of Ohio regulations (OAC-3745-9-10).

- Remove any existing solid waste including Investigative Derived Waste (IDW) from previous or current removal actions.

- Install a perimeter chain-link fence and three double-swing gates at the completion of the RA to control site access at the site in accordance with the design documents.

- Development of an Operation and Maintenance (O&M) Plan to ensure the integrity of the remedy by maintaining and repairing the concrete and asphalt cover, and the perimeter fencing for a period of thirty (30) years, and as specified in the AOC.

1.4 PROJECT OBJECTIVES AND INTENDED DATA USAGES

The primary objective of the removal action (RA) at the MMI Site is to address the lead-contaminated soils that have been determined to be a threat to human health and the environment. The RA for this site, defined in the AOC, has been designed to reduce the potential threat to human heath from lead exposure based on the intended future land use for both the site and surrounding areas. The boundaries of the RA include the 4.3-acre site and the adjacent off-site perimeter property as defined in the revised SOW.

The purpose of the data to be generated as part of this RA and covered under this QAPP is to verify that the removal performance standards for all associated RA tasks have been met in areas identified in the

revised SOW. These performance standards are discussed in the Performance Standard Verification Plan (Appendix B to the RD/RA Workplan). For this project, the tasks and associated performance standards are detailed in Section 1.3.3.

In addition, sufficient data will be gathered during project activities to verify that the performance standards associated with the short-term implementation of the RA (i.e., air sampling, any necessary wastewater or waste characterization sampling for off-site disposal, sampling of backfill material etc.) as described in the FSAP (Appendix C of the RD/RA Workplan), are met. The list of the RA activities and intended data usage is presented in Section 1, Table QAPP-1.

Data collected as part of the removal action will need to meet the Data Quality Objectives (DQOs) applicable for the end use of the data that was collected. As such, different data uses may require different levels of data quality. DQOs are qualitative and quantitative statements that specify the quality of results required to support decisions made during the project and have been in accordance with the Quality Objectives Interim Guidance Document (EPA QA/G-4).

The three types of DQOs identified for use at the site include the following:

- Screening (DQO Level 1): This provides the lowest data quality but the most rapid results, It will be used for field screening and health and safety monitoring at the site, and preliminary comparison to ARARs. This type of data will be used for the X-Ray Fluorescence (XRF) instrument and air monitoring equipment at the site.

- Engineering (DQO Level 3): This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include laboratory data with quick turnaround times used for screening but without full quality control documentation. This type of data will be used for backfill characterization, wastewater characterization, if needed, air monitoring, and waste characterization.

- Confirmational (DQO Level 4): This provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives and verification that performance standards have been met. This requires full analytical and data validation procedures in accordance with EPA recognized protocol. This type of data will be used for all confirmatory soil sampling and treatment verification sampling to verify that performance standards have been met.

1.4.1 Project Target Parameters

A summary of the project tasks, the associated sampling parameters and the intended data usage are presented in Section 1, Table QAPP-1. Holding time and preservation required for these samples is presented in Table FSAP-1, Appendix C of the RD/RA Workplan.

Specific tasks are described in the following sections.

1.4.1.1 Excavation of Lead-Impacted Soils

Excavation of site soils will be performed on an estimated 40 sample grids. The XRF field-screening device will be used to measure lead concentrations in soils to guide the lateral and vertical extent of the excavation in each grid. Excavation will proceed until either the RBRG of 1,000 mg/Kg has been met or

until historic slag is encountered (maximum depth), whichever comes first. Though soils will be field screened using an X-Ray Fluorescence analyzer during excavation activities, the XRF will NOT be used to verify that performance standards have been met. Utilization of this field-screening device will allow for more expedient decision-making regarding volume of material present requiring excavation, and treatment to render the material nonhazardous. This utilization will increase project efficiency. The XRF analyzer will be calibrated and compared to known standards on at least a daily basis in accordance with the standard operating procedure (SOP) for the XRF as presented in Attachment FSAP-1 of the Field Sampling and Analysis Plan.

If the XRF indicates the performance standard has been met prior to reaching the historic slag fill, a post-excavation confirmatory sample will be collected from the floor of the excavation in that grid to verify that the lead concentration is below 1,000 mg/Kg total lead RBRG. Samples will be collected in the center of each grid and submitted for laboratory analysis of total lead. A detailed discussion of the post-excavation confirmatory sampling methodology is provided in Appendix C, Field Sampling and Analysis Plan of the RD/RA Workplan. If the level of lead in the soil is confirmed by the laboratory to be below the performance standard, no further excavation in the grid will occur and the grid will be backfilled with clean fill material. If the confirmatory sample indicates that the performance standard has not been achieved, additional excavation will be conducted in that grid until either the RBRG has been met or until historic slag is encountered.

1.4.1.2 Stabilization of Lead-Impacted Soils

Treatment is required of excavated soils on-property and along the site perimeter to render the material nonhazardous prior to either filling low areas beneath the asphalt cover or off-site disposal. The soils will be treated using a treatment system and additive blend that has been determined to be effective during the Treatability Study as presented in Appendix E of the RD/RA Workplan. The soils will be treated to meet the nonhazardous criterion of <5.0 mg/L TCLP lead.

The treated soils to be disposed of off-site will be transported to an approved Subtitle D landfill facility. As defined in 40CFR 268.45(c)(1)(C), the treated soils will meet the Land Disposal Restriction (LDR) standard of 10 times the Universal Treatment Standard for the primary hazardous constituent (<7.5 mg/L TCLP lead) and any underlying hazardous constituents (UHCs) that may be identified during the waste profiling. The treated soils will also be less than the hazardous characteristic level for lead (<5.0 mg/L TCLP lead) or any other identified UHC to allow for off-site disposal as nonhazardous waste.

1.4.1.3 Backfilling

Following excavation in areas outside the asphalt or concrete cover, clean imported fill will be used to bring the site back to grade then vegetated. The backfill material will be tested prior to use. Analytical parameters are listed in Table QAPP-1. The frequency and sampling methodology for backfill sources are presented in Table QAPP-2, Field Sampling and Analysis Plan, Appendix C of the RD/RA Workplan

1.4.1.4 Air Monitoring

During removal activities, air monitoring will be performed for Total Suspended Particulate (TSP) and total lead particulate to ensure that the performance standard outlined in the SOW and the National Ambient Air Quality Standards are not exceeded. Personal and area air monitoring for lead will also be conducted to ensure worker safety. Air monitoring is also discussed in Section in Section 4.0 of the

FSAP (Appendix C of the RD/RA Workplan) and Section 7.0 of the HASP.

1.4.1.5 Waste Characterization

Based on the actual volume of stabilized soils that will need to be placed beneath the cap, some soils may be transported off-site for disposal as nonhazardous waste at an approved Subtitle D landfill facility, in accordance with the Final Design. In accordance to the SOW, and described in Section 1.4.1.2, contaminated soils deemed to be hazardous will be treated to not only meet the LDR standard of 10 times the Universal Treatment Standard (or 7.5 mg/L TCLP lead) as defined in 40CFR 268.45(c)(1)(C), but also to be less than the hazardous characteristic lead level (<5.0 mg/L TCLP lead) to allow for off-site disposal as nonhazardous waste. Therefore contaminated soils requiring treatment will be stabilized to nonhazardous levels (< 5.0 mg/L) using the TCLP test to measure compliance, and shipped off-site for disposal in an approved Subtitle D landfill.

Construction debris associated with demolition of above-ground concrete structures will be pressure-washed and disposed of off-site at an approved facility. Any other investigative-derived waste will be disposed in accordance to all applicable federal and state requirements.

1.4.1.6 Wastewater Characterization

Any bulked decontamination water or water pumped from excavation areas or open pits that is not used for dust control measures will be tested for applicable Northeast Ohio Regional Sewer District (NEORSD) analytical parameters to allow for discharge to the sewer system with approval from the NEORSD.

1.4.2 Field Parameters

During the implementation of the RA, XRF field screening for lead will be conducted to guide the depth of excavations. Other various field-monitoring activities will be conducted to collect information regarding worker health and safety and to evaluate the effectiveness of fugitive dust controls at the site.

Air monitoring will be conducted within the work area and along the perimeter of the work area. The air monitoring locations will be established based on wind and weather data collected on a daily basis. Air monitoring and sampling will be performed as described in the Field Sampling and Analysis Plan (Appendix D of the RD/RA Workplan).

Acceptable limits of field instrument screening errors are presented in Section 8, Table QAPP–8.

1.4.3 Laboratory Parameters

The primary purpose of the RA data collection is to gather sufficient information to verify that the performance standards outlined in the PSVP have been achieved. These standards include the RBRG for total lead in soils of 1,000 mg/Kg or the presence of historic slag, whichever is encountered first, and a treatment standard of <5.0 mg/L TCLP lead to render the excavated material nonhazardous waste. A summary of the laboratory parameters for each task and the associated QC samples are provided in Section 3.0, Table QAPP-2.

The detailed design of each sampling program, procedures and methods that will be used to acquire the data for air and soils is presented in Appendix C, Field Sampling and Analysis Plan of the RD/RA Workplan.

Acceptable limits on decision errors used to establish the sampling results are provided in Attachment QAPP-C.

1.5 SAMPLE NETWORK DESIGN AND RATIONALE

Total lead analyses will be used as the indicator for contaminant removal and surficial and subsurface soils at the site. Previous sample results from this site, coupled with experience from similar sites, indicate that not only is lead the predominant contaminant, it is a good general indicator of removal of other metals that may be co-located at the site.

Air monitoring parameters were chosen based on known contaminants and the nature of the work. Since excavation activities will be taking place, airborne contaminants are the major concern.

Table QAPP-2 in Section 3.0 of the QAPP summarizes the project samples to be taken by task, the matrix to be analyzed, the parameters to be analyzed, and the frequency of collection. Project specific reporting limits are presented in Section 7.0, Tables QAPP-3 through QAPP-7.

1.6 PROJECT SCHEDULE

The removal activities as described in the RD/RA Workplan will require approximately six weeks to complete. Refer to the Figure 3 of the RD/ RA Workplan for a detailed schedule of specific tasks.

TABLE QAPP-1
Intended Data Usage

ACTIVITY	DESCRIPTION	PARAMETERS	INTENDED DATA USAGE
Perimeter Air Monitoring	Air	Lead , TSP	Health monitoring Monitor fugitive lead and particulate emissions on-site and perimeter
Lead-Impacted Soils	Soil	XRF Lead Total Lead	Determine the vertical and horizontal extent of lead impacted soils until either the RBRG of 1,000 mg/Kg lead is met or until historic slag is encountered, whichever comes first.
Excavated soil treatment	Stabilized lead-impacted soils	TCLP Lead	Verify the treatment standards for contaminated lead-impacted soil (7.5 mg/L) are met and ensure material is rendered nonhazardous (< 5.0 mg/L) for on-site placement and consolidation.
Backfill Material Sampling	Soil (Imported Fill)	8 RCRA Metals VOCs Pesticides/PCBs TPH	Characterize imported fill material prior to use as backfill in excavated areas.
Waste Characterization Sampling for Disposal	Stabilized Soils	Waste Profile Parameters requested by Landfill	Characterize waste for off-site disposal to a nonhazardous Subtitle D Landfill facility
Wastewater Characterization Sampling for Disposal, if necessary	Bulked Wastewater	NEORD's Discharge Parameter List	Characterize wastewater to determine if it can be discharged to the city sewer system.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 PROJECT ORGANIZATIONAL CHART

Figure 2-1 of the RD/RA Workplan illustrates the lines of authority of the Removal Action Management Team for overseeing and implementing the required removal activities at the MMI site in Cleveland, Ohio. ENTACT's assigned management team may change during implementation of the RA. If there is a change in personnel of ENTACT's management team, the modification will be communicated to US EPA's RPM and the Project Coordinator.

2.2 MANAGEMENT RESPONSIBILITIES

USEPA CERCLA Project Manager, Gwen Massenberg

The USEPA CERCLA Project Manager has the overall responsibility for all phases of the Remedial Action Workplan.

Project Coordinator, Terry Casey, Efficasey Environmental LLC

The Project Coordinator's prime responsibility will be to ensure proper coordination among various project stakeholders. These stakeholders include the USEPA, OEPA, City of Cleveland, NOLTCO, Bredt & Zanick, LLC, the Project Manager, and the Respondents to the Order.

Project Manager, Mike Stoub, ENTACT.

Mr. Stoub will have the overall responsibility for ensuring that the remedial activities are implemented and completed in accordance with the AOC, revised Statement of Work, the U.S. EPA-approved RD/RA Workplan and federal, state, and local regulations. Specific responsibilities of the Project Manager will include, but not be limited to, the following:

- Providing personnel and equipment for remedial activities;
- Ensuring the RA is completed with the approved schedule;
- Ensuring effective communications between the Project Coordinator and U.S. EPA's RPM;
- Ensure that all documents and reports that ENTACT is required to generate meets the requirements of the approved workplan;
- Communicate any request for modifications to the approved workplan to the Project Coordinator and U.S. EPA; and
- Promptly notifying the Project Coordinator and U.S. EPA's RPM in the event of unforeseen field conditions and/or problems are encountered.

Field Project Manager, Bob Ainslie, ENTACT~~, Inc.~~

Mr. Ainslie will work with the Project Manager in overseeing the removal activities at the site and ensuring that the site activities are implemented and completed in accordance with the AOC, Statement of Work, the

U.S. EPA-approved RA Workplan and federal, state, and local regulations. Specific responsibilities of the Project Coordinator will include, but not be limited to, the following:

- Providing the Project Manager and USEPA's RPM the names and qualifications of contracted laboratory, disposal facilities, recycling facilities, and transporters used to implement the RA;
- Ensuring that ENTACT's associates perform their designated duties in accordance with the Health and Safety Plan;
- Ensuring required quality assurance/quality control procedures are properly implemented and documented;
- Notifying appropriate personnel identified in the Health & Safety Plan in the event of spills or air releases that exceed criteria;
- Working with the Project manager in ensuring the RA is completed following the approved schedule;
- Notifying appropriate personnel identified in the Health & Safety Plan in the event of spills or air releases that exceed criteria;
- Communicating any request for modifications to the approved workplan to the Project Coordinator and USEPA; and
- Promptly notifying the Project Manager and the USEPA's RPM in the event of any unforeseen field conditions and/or problems that are encountered.

Regulatory/Technical Leads, Pat Vojack, P.G., Mark Waxali P.E., ENTACT~~ & Associates LLC~~

Ms. Vojack and Mr. Waxali will provide regulatory, technical and engineering support to the Project Manager in ensuring that the site activities are implemented and completed in accordance with the AOC, SOW, the U.S. EPA-approved RA Workplan and federal, state, and local regulations. They will also provide technical support to the Field Manager in the areas of wastewater management and treatment, solid and hazardous waste management, air and groundwater monitoring, and any other technical design requirements for the RA.

Corporate Health and Safety Director, Mr. Jonathan Patlak, ENTACT~~ & Associates LLC.~~

The Corporate Health and Safety Officer will coordinate and provide oversight for the Health and Safety issues at the site. He will be responsible for conducting the Health and Safety Orientation meeting before the RA is implemented. He will review weekly health and safety updates from the site and conduct several inspections at the site during the RA.

Management Control Process

The ENTACT Project Manager has overall responsibility for successfully completing the remedial action at the site. This includes safely completing technical Statement of Work items, fulfilling contractual obligations, compliance with the approved workplan, and meeting all or exceeding the established project schedule and budget. The Project Manager will accomplish these objectives by monitoring the work progress, reviewing and planning each project task with experienced technical staff and the Field Project Manager, and ensuring the appropriate and sufficient resources are available to the Field Project Manager and the On-Site QA/QC Officer.

The Project Manager will receive daily progress reports from site personnel appraising him of the status of planned, ongoing, and completed work, including QA/QC performance and health and safety, site-specific issues. In addition, the Project Manager will be apprised of any potential problems and recommendations for solutions and/or corrective action.

Qualifications and experience of ENTACT's Management Team are provided in Attachment QAPP-A of the QAPP.

2.3 QUALITY ASSURANCE RESPONSIBILITIES

US EPA Region 5 Superfund's Quality Assurance Coordinator

U.S. EPA Superfund Quality Assurance Reviewer has the responsibility to review and approve all Quality Assurance Project Plans. In addition, the U.S. EPA Quality Assurance Coordinator is responsible for conducting external performance and system audits of the laboratory and evaluating analytical field and laboratory procedures.

Quality Assurance Manager, Patricia Vojack, P.G., ENTACT ~~& Associates LLC~~

The ENTACT QA Manager will be responsible for ensuring that all ENTACT procedures for this project are being followed. In addition, the ENTACT QA Manager will be responsible for the data validation of all sample results from the analytical laboratory. Specific responsibilities will include, but are not limited to, the following activities:

- Ensuring required quality controlled testing is performed and documented and the results are provided to the ENTACT's project management team, the Project Manager, and U.S. EPA in accordance with the requirements of the approved workplan;
- Providing oversight and direction to the on-site quality assurance official; and,
- Providing assistance in the modification of QA methodology or implementation based on conditions encountered during the remedial activities; if different than specified in the approved QAA.

On-Site QA Officer, Field Engineer, ENTACT ~~& Associates LLC.~~

The on-site QA officer will be responsible for performing required quality control testing at the site. The on-site Quality Control Officer will operate independently of ENTACT's Project Manager and Field Project Manager. The QA/QC Officer will communicate any QA/QC issues related to the site to the Project Manager. The QA/QC officer will have the authority to correct and implement additional measures to assure compliance with the approved workplan, including the QAPP. Specific responsibilities will include:

- Adhere to the approved QAPP;
- Document any deviations to the plan with a justification for the deviations, and if necessary appropriate notification in accordance with the approved workplan;
- Secure necessary sampling tools, bottles, packaging/shipping supplies, chain-of custody documents, etc. in accordance with the approve workplan;
- Collect or direct the collection and ship samples at the frequencies and for laboratory analysis parameters

specified in the QAPP;
- Document the location, time, and date of all samples that are collected and shipped to the laboratory;
- Interface with the superintendents such that the sample collection is coordinated with the general progression of the work;
- Notify the project manager, project coordinator and the U.S. EPA of any sampling activities associated with the implementation of the approved workplan; and
- Obtain analytical results and reporting the data to the Project Manager, Project Coordinator, and U.S. EPA's RPM.

2.4 LABORATORY RESPONSIBILITIES

The laboratories which will be performing the sample analysis for this project, except for air samples, is:

GeoAnaltyical, Inc.
9263 Ravenna Road
Twinsburg, OH 44087
Phone (330) 963-6990

The laboratory performing the air monitoring analysis is:

Pace Analytical Services, Inc.
7726 Moller Road
Indianapolis, IN 46268
Phone (317) 875-5894

GeoAnalytical Project Manager, Amy Onest

The GeoAnalytical Project Manager will report directly to the ENTACT QC Manager and will be responsible for ensuring that all resources of the laboratory are available on an as required basis. She is also responsible for the overview of final analytical reports.

GeoAnalytical Quality Assurance Officer, Terrence M. Harper

The Quality Assurance Officer has the overall responsibility for data after it leaves the laboratory. The GeoAnalytical QA Officer will communicate data issues through the GeoAnalytical Project Manager. In addition, the GeoAnalytical QA Officer will overview laboratory quality assurance and QA documentation, conduct detailed data review, determine whether to implement corrective action, and define appropriate laboratory procedures.

GeoAnaltyical Sample Custodian

The GeoAnalytical Sample Custodian will report to the GeoAnalytical Project Manager. The GeoAnalytical Sample Custodian responsibilities will include: receiving, recording and inspecting the incoming samples; verifying chain-of-custody and its accuracy; notifying laboratory manager and supervisor of sample receipt and inspection; assigning a unique identification number and customer number, and entering each into the

sample receiving log; and transferring samples to the appropriate lab section.

GeoAnalytical Technical Staff

The GeoAnalytical Technical Staff will be responsible for sample analysis and identification of corrective actions.

Qualifications and experience of GeoAnaltyical Inc. QA/QC Management Team are provided in Attachment QAPP-A of the QAPP.

Pace Analytical Project Manager, Jill Kofoed

The Pace Analytical Project Manager will report directly to the ENTACT QC Manager and will be responsible for ensuring that all resources of the laboratory are available on an as required basis. She is also responsible for the overview of final analytical reports.

Pace Analytical Quality Assurance Officer, Tim Harrison

The Quality Assurance Officer has the overall responsibility for data after it leaves the laboratory. The Pace Analytical QA Officer will communicate data issues through the Pace Analytical Project Manager. In addition, the Pace Analytical QA Officer will overview laboratory quality assurance and QA documentation, conduct detailed data review, determine whether to implement corrective action, and define appropriate laboratory procedures.

Pace Analytical Sample Custodian

The Pace Analytical Sample Custodian will report to the Pace Analytical Project Manager. The Pace Analytical Sample Custodian responsibilities will include: receiving, recording and inspecting the incoming samples; verifying chain-of-custody and its accuracy; notifying laboratory manager and supervisor of sample receipt and inspection; assigning a unique identification number and customer number, and entering each into the sample receiving log; and transferring samples to the appropriate lab section.

Pace Analytical Technical Staff

The Pace Analytical Technical Staff will be responsible for sample analysis and identification of corrective actions.

3.0 QUALITY ASSURANCE (QA) OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results, which are legally defensible in a court of law. The purpose of implementing these procedures is to assess the data generated for accuracy, precision, representativeness, completeness, and comparability for both the laboratory analytical program and field sample collection activities. The primary goal of the program is to ensure that the data generated are representative of environmental conditions at the site. To obtain this goal, a combination of statistical procedures and qualitative evaluations will be used to check the quality of the data.

Precision, accuracy, representativeness, completeness, and comparability (PARCC) will be computed in the manner described in the following paragraphs. A qualitative assessment of PARCC factors will be made and will be documented. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventative maintenance of field equipment, and corrective action are described in other sections of this QAPP.

3.1 PRECISION

The precision of laboratory results and field sampling efforts will be evaluated by examining laboratory and field QC sample results. Analytical precision will be evaluated for analytical methods by comparing the QC criteria stipulated in the standard operating procedures to the results from laboratory matrix spike/matrix spike duplicate samples and field duplicate samples.

3.1.1 Definition

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, usually expressed in terms of the standard deviation.

3.1.2 Field Precision Objectives

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 investigative analytical samples.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) for replicate samples. The equations to be used for precision in this project can be found in Section 12 of this QAPP. Precision control limits are ~~given~~provided in ~~tables in Section 8~~Attachment QAPP-C.

3.2 ACCURACY

The accuracy of the analytical data will be assessed by examining the results obtained from the analysis of sample blanks, duplicate samples, laboratory matrix spike/matrix spike duplicate samples, and method

required laboratory QA/QC samples. One equipment blank will be prepared and documented for every 10 investigative samples. One matrix spike, and one matrix spike duplicate will be analyzed for every 20 investigative samples. Data will be qualified in accordance with the appropriate EPA functional guidelines for evaluating data if either field QC blanks or laboratory QC blanks indicate that the accuracy or precision of analytical results is compromised. Field blanks will only be collected if disposable sampling equipment is used to verify that decontamination procedures are adequate ad not biasing data. It is not anticipated that any disposable sampling equipment will be used.

3.2.1 Definition

Accuracy is the degree of agreement of a measurement with an accepted reference or true value.

3.2.2 Field Accuracy Objectives

Accuracy in the field is assessed through the use of field blanks and adherence to all sample handling, preservation, and holding times.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of matrix spikes (MS) or standard reference materials (SRM) and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 12 of this QAPP. Accuracy control limits are provided in Attachment QAPP-C of the QAPP.

3.3 COMPLETENESS

3.3.1 Definition

Completeness is the amount of valid data obtained from a measurement system compared to the amount that was expected and required to meet the project data goals.

3.3.2 Field Completeness Objectives

Field completeness is the measurement of the amount of valid measurements obtained from all the measurements taken in the project. The intent of this program is to attempt to achieve a goal of 100 percent completeness. Realizing that under normal conditions this goal may not be achievable, the completeness goal for this program is 90 percent. ~~Residential well sampling completeness will be 100%.~~ This completeness goal is considered adequate to meet the data quality objectives for this site based on prior consideration of PARCC parameters, the sampling design plans, and data collection activities proposed for each medium. In developing the sampling design plan, critical data points were carefully considered and identified to help ensure comparability of data. The equation for completeness is presented in Section 12 of this QAPP.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The intent of this program is to attempt to achieve a goal of 100 percent completeness. Realizing that under normal conditions this goal may not be achievable, the completeness goal for this program is 90 percent. Residential well sampling completeness will be 100%. The laboratory equation for completeness is presented in Section 12 of this QAPP.

3.4 REPRESENTATIVENESS

Representativeness expresses the degree to which sample data accurately and precisely represent environmental conditions and parameter variations at a sampling location. Representativeness is a qualitative parameter most concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by assuring that sampling locations are properly selected and a sufficient number of investigative samples are collected.

3.4.1 Definition

Representativeness is the selection of analytical methods and sampling protocols and locations such that results are representative of the media being sampled and conditions being measured.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Field Sampling and Analysis Plan (FSAP) is followed and that proper sampling techniques are used.

3.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample-holding times, and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions. During the development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting, and constraints inherent to the RA Workplan. The rationale of the sampling network is discussed in detail in the RA Workplan and Section 4 of this QAPP.

3.5 COMPARABILITY

Comparability cannot be ensured through use of standard methods and protocols alone. In order to compare data, various important elements will be considered. During this project, three elements will be evaluated for data comparability. These three elements include analytical methods, quality of data, and sampling design. If after the initial evaluation, data do not appear comparable, the QA Manager will attempt to identify other components possibly affecting comparability, including but not limited to field conditions, sampling protocols, and the occurrence of true data anomalies.

3.5.1 Definition

Comparability is an expression of the confidence with which one data set can be compared to another.

3.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSAP is followed and that proper sampling techniques are used.

3.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented. Similar QA objectives will be used throughout the project to ensure comparability.

3.6 LEVEL OF QUALITY CONTROL EFFORT

Field blank, duplicate, and matrix spike samples will be analyzed to assess the quality of data resulting from the field sampling and analytical programs.

3.6.1 Field Data

Field blanks, for water samples, consisting of distilled water used to rinse decontaminated sampling equipment will be submitted to the analytical laboratory to provide a means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the facility that may cause sample contamination. Field blanks will be collected at a frequency of 1 per 10 water samples. Also, one field blank will be prepared for every 10 investigative samples if reusable sampling equipment is used. Sampling procedures are specified in the sampling portion of the RA Workplan and Section 4 of this QAPP.

The precision and accuracy of field measurements ~~(such as pH, conductivity, etc)~~ are discussed in Section 8.1 of the QAPP and listed in Table QAPP-8.

3.6.2 Laboratory Data

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the digestion and measurement methodology. All matrix spikes are performed in duplicate and are hereinafter referred to as MS/MSD samples. One MS/MSD will be analyzed for every 20 or fewer investigative samples per sample matrix.

TABLE QAPP-2

SUMMARY TABLE OF GRID SAMPLING AND ANALYSIS PROGRAM FOR THE REMOVAL ACTION MASTER METALS, INC. SITE, CLEVELAND, OHIO

Sample Type	Analysis Parameters	Investigative Samples			Field Quality Control Samples									Totals
					Field Duplicates			Field Blanks[4]			MS/MSD[1]			
		No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	Total
Post-Excavation Sampling	Laboratory ---- Total Lead	40	1	40	4	1	4	4	1	4	--	--	--	48
	Field ---- XRF Lead	160	1	160	--	--	--	--	--	--	--	--	--	160
Treatment Confirmation Sampling[5]	Laboratory ---- TCLP Lead	20	1	20	--	--	--	--	--	--	--	--	--	220
Backfill Testing[3]	Laboratory --- RCRA Metals[2]	2	1	2	--	--	--	--	--	--	--	--	--	5
	Laboratory --- VOCs	2	1	2	--	--	--	--	--	--	--	--	--	5
	Laboratory --- TPH [8]	2	1	2	--	--	--	--	--	--	--	--	--	5
	Laboratory --- Pesticides/PCBs	2	1	2	--	--	--	--	--	--	--	--	--	5
Waste Characterization Sampling[6]	Laboratory ---- TCLP Lead	1+	1	1+	--	--	--	--	--	--	--	--	--	1+
Wastewater Sampling[7]	NEORSD Discharge Parameters	4+	1	4+	--	--	--	--	--	--	--	--	--	4+
Air Monitoring	TSP-total lead Respirable Dust	Refer to FSAP, Table FSAP-1 for air sampling frequencies and type												

NOTES:

1 For metals analysis, no extra sample volume is required; MS/MSD will be performed at a rate of one per twenty investigative samples analyzed by the laboratory.
2 RCRA Metals = arsenic, barium, cadmium, chromium, lead, selenium, silver, and mercury.
3 Estimate of one sample to be collected for every 10,000 yards of material per source.
4 Field blank samples are only required if re-usable sampling equipment is used (i.e. stainless steel bowls or trowels)..
5 Assumes analysis of one treatment sample for every 250 cubic yards for the first 1,000 yards and every 500 yards thereafter. Assumes 1,800 to 3,600 cubic yards of material to be treated.
6 If actual volume necessitates some off-site disposal as nonhazardous special waste.
7 Bulked wastewater not used for dust suppression, will be sampled for NEORSD discharge parameters for discharge to city sewer system.
8 If the TPH results exceed the petroleum faction residual saturation concentrations listed in Table 1 under Ohio Rule 3745-300-8 (8 to 40 mg/Kg for glacial till or silty clay soils), the fill will then be analyzed for SVOC compounds.

4.0 SAMPLING PROCEDURES

This section summarizes the sample documentation, sampling procedures and the QC sample preparation requirements associated with the RA tasks. A detailed discussion of the sampling procedures is presented in the Field Sampling and Analysis Plan (FSAP), presented in Appendix C of the Final RD/RA Workplan, revision 1.

Details on holding times, sample preservation and bottle requirements are presented in the FSAP, Table FSAP-1. The holding time for pesticides/PCBs listed in Table FSAP-1 reflects the post-extraction holding time of 40 days. However, pesticide and PCB samples also have a pre-extraction holding time requirement of fourteen days

4.1 SAMPLE DOCUMENTATION/IDENTIFICATION

The designated sample identification system is discussed in detail in Section 2.2 of the FSAP and summarized below:

Samples Type	Identification System
Air Samples:	
TSP High Volume Samples	TSP-Unit#-001
Personal/Area Low Volume Samples	PAS-Unit#-001
Soil Samples:	
X-Ray Fluorescence Field Screening	X-01-1
Post-Excavation Confirmatory Samples	V-01-2.0'
Treated Material-Confirmation (TCLP) Samples	TS-001
Imported Backfill Samples	BF-001
Waste Characterization Samples:	
Solid Waste (stabilized soils, if needed)	W-001
Wastewater	WW-001
Quality Control Samples:	
Field Duplicate Samples for Soil, and Treated Material	V-01-2.0'D TS-001D
Field Rinsate Blanks	FB-001

Sample identification documents will be carefully prepared to maintain identification and chain-of-custody records, and to control sample disposition. Components of the field documentation procedures include the use of field logbooks, sample labels, and the chain-of-custody forms. Original data recorded

Sample identification documents will be carefully prepared to maintain identification and chain-of-custody records, and to control sample disposition. Components of the field documentation procedures include the use of field logbooks, sample labels, and the chain-of-custody forms. Original data recorded in field logbooks, chain-of-custody records, and other forms will be written in waterproof ink. None of these documents will be altered, destroyed, or discarded, even if they are illegible or contain inaccuracies that require a replacement document. If an error is made on a document assigned to one individual, that individual will make the corrections by making a line through the error, entering the correct information, and initialing and dating the change. Samples and documentation will be maintained and handled by as few people as possible.

4.2 SAMPLE COLLECTION/PREPARATION PROCEDURES

Sample collection methodology is described in detail in Section 3.0 and Section 4.0 (air) of the FSAP and summarized in the following subsections.

4.2.1 XRF Field Screening

The XRF or Lead analyzer will be used on site during excavation activity only as a screening tool to assess the total lead concentration in soils but will not be used to verify that performance standards have been met. The area to be screened will be prepared by scraping the top layer of potentially cross-contaminated soil with a stainless steel trowel or plastic scoop and smoothing the area flat so as not to pierce the Mylar window of the probe. The in-situ measurement will be made by placing the XRF probe on a flat area of the ground surface and scanning the soil surface.

The particular instrument to be used is the Spectrace 9000 Portable XRF Analyzer or comparable Lead Analyzer. This device utilizes a probe, which consists of a sealed aluminum enclosure containing a high-resolution mercuric iodide detector and three radioisotope x-ray excitation sources, Fe-55, Cd-109 and Am-241. The Spectrace 9000 utilizes a fundamental parameter XRF calibration derived from theoretical considerations. The menu-driven software supports multiple XRF calibrations called "Applications". Each Application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of fundamental parameter calibration coefficients.

The Standard Operating Procedure (SOP) for the XRF instrument is included in Attachment FSAP-1 of the FSAP. The XRF field screening data may be tabulated for presentation in the final report, but is not to be used to confirm that the performance standards have been met.

4.2.2 Confirmatory Sampling

If excavation is terminated in a grid prior to reaching the historic slag (maximum depth), a confirmatory sample will need to be collected to verify that the RBRG of 1,000 mg/Kg has been met for that grid. The sample will be collected as a grab sample using the following equipment and supplies:

- Stainless steel or plastic disposable scoops or trowels
- Sample containers and plastic bags
- Field notebook

- Chain-of-custody form
- Decontamination supplies (Decontamination may be conducted at the sample location staging area or the main decontamination area)

Field notes will be recorded for each sample taken and will include sample identification, soil description (color, type, and foreign material) and any other pertinent observations relating to the sample or site conditions at the time of sampling.

The sample will be obtained by excavating soil from a depth of approximately 0 to 3 inches below excavated ground surface using either a decontaminated stainless steel trowel or a clean plastic disposable scoop. An additional quantity of sample material will be obtained at 10 percent of the sample locations for a field duplicate and will be shipped to the laboratory. The sample material will be stirred in a Ziploc plastic bag or stainless steel bowl to homogenize, then split in half to make each sample portion. Replicate/split samples will be also be provided to the EPA upon request.

4.2.3 Backfill Characterization Sampling

Backfill samples will be collected as single grab samples from the representative material for each source and for each type of material prior to shipment to the site to ensure the material meets both the chemical and geotechnical requirements and then at increments of one sample per 10,000 tons. A change in source location will require the collection of a new initial sample round for each type and source used. No field duplicates, field blanks or MS/MSD samples will be collected for the backfill samples.

The samples will be submitted to the designated Project Laboratory, GeoAnalytical, Inc., Twinsburg, Ohio, for chemical analysis of the applicable parameters using DQO Screening Level in accordance with the QAPP. DQO Screening Level ~~2~~3 will provides the appropriate level of quality assurance data for fill material characterization. Samples will also be submitted either to the selected geotechnical testing laboratory or will be tested by the source supplier with certification provided to ENTACT for review and approval.

4.2.4 Waste Characterization Sampling

Waste characterization samples will be collected as grab samples from representative material for the parameters listed in Table 1. The frequency of collection is dependent on landfill requirements as well on the RCRA classification of the material. Waste characterization sampling will follow the procedures outlined in the FSAP, Section ~~5.2.2~~3.3. No field duplicates, field blanks or MS/MSD samples will be collected for the waste samples.

The samples will be submitted to the designated Project Laboratory, Geo-Analtyical, Twinsburg, Ohio, for off-site laboratory analysis of the applicable parameters using DQO Screening Level in accordance with the QAPP. DQO Screening Level typically provides the appropriate level of quality assurance data for waste characterization.

4.2.5 Air Sampling

Two types of air samples will be collected at this site. TSP samples will be collected to determine the total quantity of dust in the air that can be entrained in the respiratory system and the amount of lead particles in the air. Personal / area air samples will be collected in order to monitor worker safety conditions as specified in the HASP. The units will be calibrated in accordance with the manufacturer recommendations.

Personal / area air samples will be obtained for personnel and areas by using battery powered Gilian HFS 513 Hi Flow Samplers or equivalent with 37 mm mixed cellulose ester filters. Personal air samples will be taken from the breathing zone of the workers. On-site area samples will be taken in areas where one could reasonably expect elevated airborne lead levels to occur during work activities. Each pump will be calibrated before and after each use using a primary standard (rising soap film). If a variation is found in the flow rate established during the pre and post sampling calibration, the lower, more conservative flow rate will be used and all volume calculations will be based upon the lower flow rate. The flow rate of all pumps will be between 2.0 and 4.0 liters per minute.

One lot blank will be provided to the laboratory per box of filters. No additional QC samples are required for air sampling.

The Standard Operating Procedures for the Total Suspended Particulate (TSP) matter, and Personal / Area Air samplers are provided in Attachments FSAP-2, and FSAP-3 of the Field Sampling and Analysis Plan.

4.3 FIELD QC PROCEDURES

Field duplicate will be collected for confirmatory soil samples and treatment verification samples at a rate of one duplicate for every ten investigative samples collected. At the designated sample location where a duplicate sample will be collected, an ample volume of material will be placed in a Ziploc plastic bag or stainless steel bowl and thoroughly homogenized prior to filling the sample jars. The field duplicate sample will be blind labeled as FD-001 and continue sequentially from 001 with the associated investigative sample recorded in the logbook.

If reusable-sampling equipment is used, (i.e. stainless steel bowl and/or trowel), a field blank sample will be prepared at a rate of one rinsate sample for every 10 investigative samples taken by pouring distilled water over the decontaminated sampling equipment.

MS/MSD samples will be performed at a rate of one for every 20 investigative samples analyzed by the laboratory. No extra sample volume is required for the MS/MSD samples for metals. The MS/MSD will be performed at a rate of one per twenty investigative samples.

4.4 SAMPLE CONTAINERS, PRESERVATIVES AND VOLUME REQUIREMENTS

Confirmatory soil samples and treatment verification samples will be placed into clean plastic or glass 2- and 4-ounce containers for soil samples and 8-ounce containers for TCLP lead analysis. Sample jars will

be supplied by a vendor or laboratory and will be certified clean. There are no preservatives required for either analyses and the container should be completely filled. The container will be labeled with the sample identification number, date and time of sampling and the initials of the sampler. The sample container will be placed in a sealed plastic bag for transportation to the laboratory. The designated laboratory will provide a daily courier service during remedial activities to allow for an expedited analytical turn-around time. If samples must be transported by means of commercial transportation, the samples will be placed in a cooler, packaged in a manner to prevent shifting and breakage in transit, and a custody seal will be placed on the cooler housing the samples such that any tampering with the cooler will be evident by the seal. No ice is required for metal parameters. Sample labels and custody seals are presented in Attachment QAPP-D.

Backfill or waste profile samples that include multiple parameters will be placed into the appropriate container specified in Table FSAP-1 of the FSAP. The volatile organic compound sample will be collected first and placed directly into the sample container to minimize any loss of volatile compounds, with no mixing or homogenizing the soils to prevent loss of potential volatiles contaminants

Sample containers and preservatives are not required for the XRF screening samples. If it is impractical to obtain an in-situ sample, then clean ziplock bags can be used as sample containers. These bags will be labeled to identify the sample identification code, date, time, and sampler's initials.

Air sample filters will be supplied by the laboratory. The sample filters will not be open, left out or tampered with prior to sampling. There are no preservatives required for lead or PM10 analysis.

4.5 SAMPLE CUSTODY

A Chain-of-Custody (COC) form will be filled out at the time of sampling. Information to be recorded on the COC includes sample identification, sample description, name(s) of sampler(s), and requested analyses. The COC will be placed in a sealed plastic bag for protection and will accompany the associated samples to the laboratory. Any time the sample custodian changes, the person relinquishing the samples shall sign the COC and note the date and time of transfer. The person receiving the samples shall also sign the COC and note the date and time of transfer. Attachment QAPP-D of the QAPP includes examples of COC forms for GeoAnalytical and Pace Analytical.~~An example GeoAnalytical COC is located in Attachment QAPP-D of the QAPP.~~

4.6 DECONTAMINATION PROCEDURES

All re-usable sampling equipment will be decontaminated utilizing a triple rinse procedure. During this procedure, the sampling equipment is scrubbed in a potable water/detergent wash (gross rinse), rinsed in potable water (intermediate rinse), and rinsed with distilled water (final rinse). All three decontamination fluids are changed as needed to ensure proper decontamination; however, to conserve the quantity of waste generated, ENTACT will downgrade the three phase fluids. For example, the final phase fluids are downgraded to intermediate fluids, intermediate fluids are downgraded to gross fluids, gross fluids are collected in a DOT approved container, and fresh distilled water is placed in the final phase. This method minimizes waste and ensures that the final phase fluids are clean. Spent decontamination fluids will be collected throughout the project for proper disposal at an authorized treatment facility.

After decontamination, the sampling equipment will be dried with disposable towels and stored in plastic sampling tool boxes between sampling events. All decontaminated equipment within the sampling tool box will be placed in individual plastic bags or wrapped in disposable towels. The sampling tool boxes will also be decontaminated weekly to ensure cleanliness. All trash and PPE generated during sampling will be placed in designated disposal containers for such items.

4.7 SAMPLE PACKAGING AND SHIPMENT PROCEDURES

Sample containers will be laboratory prepared and shipped in sealed containers to assure that they remain clean. Sample containers will be selected to ensure compatibility with the media being collected, preserve sample integrity, and minimize breakage during transportation. Sample labels will be filled out at the time of sampling and will be affixed to each container to identify sample number, sampler's name, date and time of collection, location of sampling point, and project identification data.

After the containers for a given sampling location have been filled out, they will be placed in plastic Ziplock storage bags, on ice (for VOC, SOC and pesticide/PCB samples only), in an insulated cooler, to be delivered to the analytical laboratory. *Each sample container will be secured in packing material, as* appropriate, for shipment to the designated laboratory. The insulated cooler lid will be taped closed and sealed to avoid the entrance of contaminants into the cooler and to avoid leaking from the cooler. Shipment of samples to the laboratory will take place on the same day as collection. The Chain-of-Custody form will be enclosed in a sealed plastic bag and adhered inside the sealed cooler. If the samples are sent by common carrier, a bill of lading will be used to document the custody of the sample while in transit. Commercial carriers are not required to sign the COC forms as long as the forms are sealed inside the cooler.

5.0 CUSTODY PROCEDURES

Custody is one of several factors which is necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all original laboratory reports, are maintained under document control in a secure area.

A sample or evidence file is under one's custody if:

- the item is in actual possession of a person; or
- the item is in the view of the person after being in actual possession of the person; or
- the item was in actual physical possession but is locked up to prevent tampering; or
- the item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

Sample identification documents will be carefully prepared to maintain identification and chain-of-custody records and to control sample disposition. Components of the field documentation procedures include the use of field logbooks, sample labels, and the chain-of-custody forms. Original data recorded in field logbooks, chain-of-custody records, and other forms will be written in waterproof ink. The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched.

5.1.1 Field Logbook Records

A field log of daily activities will be used to record sampling activities on a daily basis. This book will be bound and have consecutively numbered pages. Entries in the field logbook will be made in ink and will include: the name of the author; date and time of entry; location of activity; names and affiliations of personnel on site; sample collection or measurement methods; number of samples collected; daily weather report; sample identification numbers; field observation and comments; sampling depth increment for soils; field measurements; locations of photographs; and any deviations from the sampling plan. Each logbook will be assigned a project specific document number. The field log book will be stored in the job trailer when it is not in use.

5.1.2 Sample Labels

Sample labels are necessary to prevent misidentification of samples. Preprinted labels will be provided prior to the sampling activities. Each label will contain space for the following information: name of site, sample identification, date and time of sample collection, media sampled, name of sampler, preservatives, and types of analyses to be performed. Example of custody seal and label is provided in Attachment QAPP-D of the QAPP.

5.1.3 Chain-of-Custody Records

A Chain-of-Custody (COC) form will be completed to record the custody of every sample collected. A COC form will accompany every shipment of samples to the analytical laboratory in order to establish the documentation necessary to trace sample possession from the time of sample collection through sample analysis.

The sample portion of the COC form will include the following:

- Project number, name and location;
- Sample identification;
- Name of Project Manager, Sampler, and Recorder;
- Sampling information (sampling area, depth, media type, type of sample, date and time of collection, etc.)
- Analysis to be performed;
- Preservatives used, if any; and
- Signatures of persons involved in the COC possession, including dates.

When a Chain-of-Custody form is filled out, one page of the three-part form is retained and placed in a file at the on-site office. The other two parts of the form accompany the sample to the laboratory. One of those pages is retained by the laboratory and the other is returned with the sample result report. When the sample report is received, it is cross-checked with the COC file record and both COC pages and the laboratory report are placed in a file in fireproof storage at the on-site office. The analytical result is also entered into a computer database consisting of a comprehensive list of all samples taken at the site and the analytical results.

5.2 LABORATORY CUSTODY PROCEDURES

Samples, which are delivered by clients or received by courier, are placed in a secure Sample Control Area immediately upon delivery. Coolers containing samples are unpacked within ½ hour of receipt or placed in the walk-in cooler until unpacked. The COC accompanying the samples will be signed by the Sample Custodian or their designee at the time of delivery by the client, or in the case of courier delivery, where the COC is sealed up inside of the cooler, at the time of unpacking.

At the time of arrival and/or unpacking, coolers will be inspected for evidence of damage. They will be unpacked carefully and samples will be organized on the lab bench in numerical order or by sample sets and assigned a laboratory job number. The condition of both shipping containers and sample containers will be recorded on the internal COC form.

Information on the COC shipped with samples will be verified and recorded as to agreement or non-agreement. Labels will be checked for notation of proper preservation. If there is an apparent non-agreement in the document or incorrect preservation noted, the apparent problem will be recorded and the ENTACT Project Manager notified. The samples will then be marked or labeled with laboratory sample numbers. Laboratory project numbers are assigned serially, with each sample numbered as a subset of the project number. Finally, samples will be placed in appropriate storage and/or secure areas.

5.3 FINAL EVIDENCE FILES

The final evidence file will be the central repository for all documents, which constitute evidence relevant to sampling and analysis activities as described in this QAPP. ENTACT is the custodian of the evidence file and maintains the contents of the evidence files for the MMI removal action, including all relevant reports, records, logs, field notes, pictures, and data reviews in a secured, limited access area under the custody of the ENTACT Project Manager.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

Procedures described in this section pertain to the calibration, maintenance, and operation of equipment and instrumentation to be used during the implementation of the remedial action. A variety of instruments, equipment, and sampling tools will be used to collect data and samples to monitor site conditions. Proper calibration, maintenance, and use of instruments and equipment are~~is~~ imperative to ensure the quality of all data collected. A record of calibration and maintenance activities is important to provide legally dependable data.

Instruments and equipment used to gather, generate or measure environmental and physical testing data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility are consistent with the manufacturer's specifications.

6.1 FIELD INSTRUMENT CALIBRATION

All instruments and equipment purchased or used for the MMI removal action will be inspected to ensure that the item meets and performs to manufacturer's specifications and project specifications. Instruments meeting these requirements are issued to a field technician trained in instrument operation and made available for site use. All field equipment will be calibrated in accordance with the specific field SOPs located ~~in Attachment QAPP-C of the QAPP and~~ in Attachments FSAP-A, FSAP-B, and FSAP-C of the Field Sampling and Analysis Plan. All air samplers will be calibrated in accordance with manufacturer recommendations.

The XRF will be calibrated with the manufacturer's standards and three site-specific standards. Each standard and sample reading will be taken in triplicate and averaged. To check the initial calibration, the middle calibration standard will be rechecked after every twenty samples. A record of the instrument calibration will be maintained in a bound field notebook and these records will be subject to a QA audit. Information recorded will include the following:

- Date of calibration
- All data pertaining to the calibration procedures
- Initials of analyst performing calibration
- Adjustments made to equipment prior to and following calibration; and
- Record of equipment failure

Field instruments that will be used during this project include an X-Ray Fluorescence Analyzer, or comparable Lead Analyzer, TSP and personal/area air samplers.

Any items found to be inoperable will be taken out of use and a note stating the time and date of this action will be made in the calibration logs. The reason for equipment failure and the time and date of its return to service will also be noted in the logbook. Records produced shall be reviewed, maintained, and filed by the field operators. The ENTACT Project Manager will audit these records to verify complete adherence to these procedures.

6.2 LABORATORY INSTRUMENT CALIBRATION

All laboratory instrument calibration procedures can be found in the attached SOPs (Attachment QAPP-B).

7.0 ANALYTICAL AND MEASUREMENT PROCEDURES

The laboratory that will be performing all sample analysis for this project, except for air samples, is:

GeoAnalytical Inc.
9263 Ravenna Road
Twinsburg, Ohio
Phone: (330) 963-6990

Laboratory accreditations and certifications are presented in Attachment QAPP-E.

The laboratory that will be performing air analyses for this project is:

Pace Analytical Services, Inc.
7726 Moller Road
Indianapolis, IN 46268
Phone: (317) 875-5894

Complete list of analytical parameters, methods, matrices, holding times and preservation requirements are included in the FSAP, Table FSAP-1.

7.1 FIELD ANALYTICAL PROCEDURES

Field analytical and test procedures include the following:

Soil
XRF - Total Lead

The SOP for this device is located in Attachment FSAP-A of the Field Sampling and Analysis Plan, Appendix C of the RD/RA Workplan.

Air
TSP Air Monitor
Area/Personal Air Monitors

The SOP for these monitors are located in Attachment FSAP-B and FSAP-C of the Field Sampling and Analysis Plan, Appendix C of the RD/RA Workplan.

7.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory analytical test procedures include the following:

Soil:

Total Lead - Method 6010/6020

Treated Soils:

TCLP lead - Method 1311/6010

Off-Site Backfill Source:

Total RCRA Metals – Method 6010/6020/7471
TPH - Method 8015 (SVOC analysis (Method 8270C) may be required depending on TPH levels)
VOCs – Method 8260
Pesticides/PCBs – Method 8081

Air Monitors:

Total lead and particulate matter less than 10μm (PM_{10}) – Method 7082/7105/7300/Appendix G of 40 CFR 50

The air analytical results will be provided by Pace Analytical Services, Inc. of Indianapolis, IN . The SOPs for the air monitoring are provided in Attachments FSAP-B and FSAP-C of the Field Sampling and Analysis Plan. Pace Analytical's SOPs for sample handling are provided in Attachment QAPP-A.

All SW-846 methods will be used for analysis. Analytical methods and extraction methods for soil, air and backfill are provided in the FSAP, Table FSAP-1.

7.3 LIST OF TARGET COMPOUNDS AND LABORATORY REPORTING LIMITS

The reporting limits are given in Table QAPP-3 through QAPP-7 for the analyses required during the RA. The instrument detection limit is determined once per quarter and is confirmed to be less than the reporting limit. Current instrument and method detection limits are presented in the applicable SOP in Attachments QAPP-B1 through QAPP-B16.

TABLE QAPP-3
Total Metals
Method 6020/7471A Soil Limits

Metal	Matrix	Method	Reporting Limit (mg/Kg)
Arsenic (ICAP)	Soil	SW-6020	5.0
Barium (ICAP)	Soil	SW-6020	5.0
Cadmium (ICAP)	Soil	SW-6020	1.0
Chromium (ICAP)	Soil	SW-6020	2.0
Mercury (CVAA)	Soil	SW-7471A	0.10
Selenium (ICAP)	Soil	SW-6020	5.0
Silver (ICAP)	Soil	SW-6020	1.0
Lead (ICAP)	Soil	SW-6020	1.0

TABLE QAPP-4
Volatile Organic Compounds
Method 8260 Soil Limits

Compound	Method Reporting Limit (µg/kg)
Dichlorodifluromethane	5
Chloromethane	5
Vinyl chloride	5
Bromomethane	5
Chloroethane	5
Trichlorofluromethane	5
1,1-Dichloroethene	5
Trans-1,2-Dichloroethene	5
Methyl-tert-butyl ether	5
1,1-Dichloroethane	5
2,2-Dichloropropane	5
cis-1,2-Dichloroethene	5

TABLE QAPP-4 continued
Volatile Organic Compounds
Method 8260 Soil Limits

Compound	Method Reporting Limit (µg/kg)
Bromochloromethane	5
Chloroform	5
1,1,1-Trichloroethane	5
Carbon Tetrachloride	5
1,1-Dichloropropene	5
Benzene	5
1,2-Dichloroethane	5
Trichloroethene	5
1,2-Dichloropropane	5
Dibromomethane	5
Bromodichloromethane	5
cis-1,3-Dichloropropene	5
Toluene	5
Trans-1,3-Dichloropropene	5
1,1,2-Trichloroethane	5
1,3-Dichloropropane	5

TABLE QAPP-4 continued
Volatile Organic Compounds
Method 8260 Soil Limits

Compound	Method Reporting Limit (µg/kg)
Tetrachloroethene	5
Dibromochloromethane	5
1,2-Dibromomethane	5
Chlorobenzene	5
1,1,1,2-Tetrachloroethane	5
Ethylbenzene	5
Total Xylenes	5
Styrene	5
Bromoform	5
Isopropylbenzene	5
Bromobenzene	5
1,1,2,2,-Tetrachloroethane	5
1,2,3-Trichloropropane	5
n-Propylbenzene	5
2-Chlorotoluene	5
4-Chlorotoluene	5
1,3,5-Trimethylbenzene	5

TABLE QAPP-4 continued
Volatile Organic Compounds
Method 8260 Soil Limits

Compound	Method Reporting Limit (µg/kg)
tert-Butylbenzene	5
1,2,4-Trimethylbenzene	5
sec-Butylbenzene	5
1,3-Dichlorobenzene	5
p-Isopropyltoluene	5
1,4-Dichlorobenzene	5
1,2-Dichlorobenzene	5
n-Butylbenzene	5
1,2-Dibromo-3-chloropropane	5
1,2,4-Trichlorobenzene	5
Hexachlorobutadiene	5
Naphthalene	5
1,2,3-Trichlorobenzene	5
Acetone	25
2-Butanone	25
Carbon Disulfide	5
2-Hexanone	25
4-Methyl-2-pentanone	25

TABLE QAPP-5
Total Petroleum Hydrocarbons (TPH)
Method 8015

Compound	Matrix	Method Reporting Limit (mg/Kg)
TPH [see note]	Soil	4

Note: Backfill material will be sampled for TPH. If TPH levels exceed the petroleum faction residual saturation concentrations listed in Table I under Ohio Rule 3745-300-8 (8 to 40 mg/Kg for glacial till to silty clay soils) the fill material will then be sampled for semi-volatile organic compounds as listed below.

TABLE QAPP-6
Semi-Volatile Organic Compounds (SVOCs)
Method 8270

Parameter	Method Reporting Limit (µgg/Kg)
Acenaphthene	330
Acenaphthylene	330
Anthracene	330
Benzo(a)anthracene	330
Benzo(b)fluoranthene	330
Benzo(k)fluoranthene	330
Benzo(a)pyrene	330
Benzo(g,h,i)perylene	330
Benzyl alcohol	330
Bis(2-ethylhexyl)phthalate	330
Chrysene	330
Dibenzo(a,h,i)anthracene	330
Dibenzofuran	330
Di-n-butylphthalate	330

TABLE QAPP-6 continued
Semi-Volatile Organic Compounds (SVOCs)
Method 8270

Parameter	Method Reporting Limit (µg/Kg)
1,2-dichlorobenzene	330
1,4-dichlorobenzene	330
2,4-dichlorophenol	330
2,4-dimethylphenol	330
2,4-dinitrotoluene	330
2,6-dinitrotoluene	330
Di-n-octylphthalate	330
Fluoranthene	330
Fluorene	330
Hexachlorobenzene	330
Hexachlorobutadiene	330
Hexachlorocyclopentadiene	330
Hexachloroethane	330
Indeno(1,2,3-cd)pyrene	330
Isophorone	330
2-Methylnaphthalene	330
2-Methylphenol	330
4-Methylphenol	330
Naphthalene	330
2-Nitroaniline	330
4-Nitroaniline	330
Nitrobenzene	330
2-Nitrophenol	330

TABLE QAPP-6 continued
Semi-Volatile Organic Compounds (SVOCs)
Method 8270

Parameter	Method Reporting Limit (µg/Kg)
4-Nitrophenol	330
Pentachlorophenol	330
Phenanthrene	330
Phenol	330
Pyrene	330
Carbazole	330
1,2,4-Trichlorobenzene	330
2,4,6-Trichlorophenol	330
2,4-Dinitrophenol	1650
Diethylphthalate	330
4-Chlorophenyl-phenyl ether	330
4.6-Dinitro-2-methylphenol	330
4-Bromophenyl-phenyl ether	330
Butylbenzylphthalate	330
3,3-Dichlorobenzidine	1650
bis(2-Chloroethyl) ether	330
2-Chlorophenol	330
1,3-Dichlorobenzene	330
N-Nitrosodipropylamine	1650
bis(2-Chloroethoxy)methane	330
4-Chloroaniline	330
4-Chloro-3-methylphenol	330
2,4,5-Trichlorophenol	330

TABLE QAPP-6 continued
Semi-Volatile Organic Compounds (SVOCs)
Method 8270

Parameter	Method Reporting Limit (µg/Kg)
2-Chloronaphthalene	330
2-Methyl-4,6-dinitrophenol	1650
Dimethyl phthalate	330
3-Nitroaniline	330

TABLE QAPP-7
Pesticides/PCBs
Method 8081 Soil Limits

Compound	Method Reporting Limit (µg/Kg)
Aldrin	0.05
Alpha-BHC	0.05
Beta-BHC	0.05
Delta-BHC	0.05
Chlordane	85
4,4'-DDD	3.3
4,4'-DDE	3.3
4,4'-DDT	3.3
Dieldrin	3.3
Endosulfan I	1.7
Endosulfan II	3.3
Endosulfan Sulfate	3.3
Endrin	3.3
Endrin Aldehyde	3.3
Heptachlor	1.7
Heptachlor epoxide	1.7
Lindane	1.7
Methoxychlor	17
Toxaphene	85
Aroclor 1016	33
Aroclor 1221	33
Aroclor 1232	33
Aroclor 1242	33
Aroclor 1248	33
Aroclor 1254	33
Aroclor 1260	33

8.0 QUALITY CONTROL CHECKS

Internal QC procedures are designed to ensure and document the overall quality of data. Two types of QC checks will be employed to evaluate the performance of the laboratory's analytical procedures. The QC checks represent the system checks and controlled samples introduced into the sample analysis stream that are used to validate the data and calculate the accuracy and precision of the chemical analysis program.

Project QC checks are accomplished by submitting controlled samples into the laboratory from the field. Two external types of QC samples will be used: blanks and duplicates. A duplicate sample will be collected for every 10 samples per matrix or one duplicate per day, whichever is greater. Any samples submitted as "blind" samples will be noted in the field logbook and given a sample number that does not indicate to the laboratory that the sample is a QC check.

8.1 FIELD QUALITY CONTROL CHECKS

For field XRF soil analyses, a laboratory sample will be sent to the laboratory for confirmatory total lead analysis for ten percent of the investigatory samples. Table QAPP-8 presents the QA criteria for field measurements.

8.2 LABORATORY QUALITY CONTROL CHECKS

Laboratory QC checks are accomplished through the use of system checks and QA/QC samples that are introduced into the same analysis stream. Laboratory system checks and QA/QC samples for inorganics are defined below.

- Calibration Blank - A volume of acidified de-ionized water.
- Continuing Calibration - Analytical standard run every 10 analytical samples or every two hours, whichever is more frequent, to verify the calibration of the analytical system.
- Instrument Calibration - Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity, and dynamic range of the instrument to target compounds.
- Preparation Blank - An analytical control that contains deionized water and reagents, carried through the entire analytical procedures. An aqueous method blank is treated with the same reagents as a sample with a water matrix; a solid method blank is treated with the same reagents as a soil sample.

Laboratory QA/QC checks will be performed and samples will be analyzed at a frequency established by appropriate SW-846 protocols for inorganic compounds and appropriate SOPs for analytical methods. Attachment QAPP-C defines all the GeoAnaltyical, Inc. QC check criteria for this project. Any QC checks that do not meet acceptance criteria will be handled as discussed in Section 13.0 of the QAPP.

Table QAPP-8
FIELD QC CRITERIA

PARAMETER	METHOD[1] REFERENCE	PRECISION[2]	ACCURACY[2]	COMPLETENESS
SOIL				
Field XRF	Per ENTACT SOP	± 20%	N/A[3]	90%

NOTES:

1. Methods: E - *Method for Chemical Analysis for Water and Wastes* (U.S. EPA, 1983).
   SW-xxxx – *Methods for the Analysis of Solid Waste (SW-846).*
2. Acceptable accuracy and precision based on the range of measurement. The XRF will be used for screening purposes only and to guide depths of excavation during remedial activities. Laboratory confirmation samples will be the determining factor as to whether cleanup criteria is achieved.
3. NA - Not Applicable

9.0 DATA REDUCTION, VALIDATION AND REPORTING

All data collected will be managed, distributed, and preserved to substantiate and document that data are of known quality and are properly maintained. Technical data will be tracked and validated to monitor the performance of the tasks. An outline of the QC data handling process for data collection, transfer, validation, reduction, reporting, and storage for both field and laboratory QC data is described below. The ENTACT QA Manager is responsible for these tasks.

9.1 DATA REDUCTION

Data quality and utility depends on many factors, including sampling methods, sampling preparation, analytical methods, quality control, and documentation. Once all physical and chemical data are validated and assembled, these data are further evaluated with respect to precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters. Satisfaction of these criteria will be documented as listed below. Chemical data must meet criteria of (1) quantitative statistical significance, (2) custody and document control, and (3) sample representativeness. Physical data must meet criteria of: (1) sampling location, time, and personnel; (2) documentation; and (3) methodologies.

To determine the quantitative statistical significance of chemical data, the following items will be documented as appropriate:

- Laboratory/field instrumentation, including calibration data, standard methods, and references;
- Proper sample bottle preparation;
- Laboratory analysis detection limits;
- Analysis of laboratory (reagent) blanks at a frequency of at least one per 20 samples per matrix;
- Analysis of laboratory spikes at a frequency of at least 1 per 20 samples or one per analytical batch;
- Analysis of field replicates (duplicates or splits) at a frequency of at least 1 per 10 samples for each matrix or one per day, whichever is greater;
- Analysis of laboratory replicates (duplicates or splits) at a frequency of at least 1 per 20 samples;
- Presentation of tabulated QC data; and
- QA/QC certification of the laboratory is semi-annually through the Ohio EPA Voluntary Action Program and annually through the Ohio Drinking Water program for VOCs only.

To evaluate the custody and document control for samples and results, the following items will be documented:

- Field custody noted in field logbook or chain-of-custody documentation available;
- Samples either couriered or hand-delivered to laboratory with chain-of-custody documentation available;
- Laboratory custody documented by chain-of-custody documentation from either field personnel or shipper;
- Laboratory custody documented through designated laboratory sample custodian with secured sample storage area;
- Sample designation number(s) traceable through entire laboratory monitoring system;
- Field notebooks and all custody documents stored in secure repository or under the control of a document custodian;
- All forms filled out completely in indelible ink without alterations except as initials;
- Identity of sampler; and
- Date of sample collection, shipping, and laboratory analysis.

To determine sample representativeness the following items must be checked:

- Compatibility between appropriate field and laboratory measurements or suitable explanation of discrepancy;

- Analysis within holding time limits suitable for the preservation and analysis methods used;
- Sample storage within suitable temperature, light, and moisture conditions;
- Proper sample containers used;
- Proper sample collection equipment used and properly decontaminated;
- Proper sample preservation;
- Proper laboratory preparation techniques used;
- An evaluation of factors to determine bias screening; and
- Sample site selection criteria to provide representativeness.

To evaluate the field physical data that support the analytical data, the following items will be documented:

- Sampling date and time;
- Sampling personnel;
- Sampling location;
- Physical description of sampling location;
- Sample collection technique;
- Field preparation techniques;
- Visual classification of sample using an accepted classification system;
- A thorough description of the methodology used and a rationale for the use of that methodology;
- Complete documentation of record-keeping practices;
- Field notebook and all custody documents stored in a secure repository or under the control of a document custodian; and
- All forms filled out in indelible ink without alterations except as initialed.

9.1.1 Field Data Reduction Procedures

Field data reduction is not anticipated for this project. The data will be generated from direct readout instruments. The data is then downloaded by RS-232 computer port to a database spreadsheet. The field XRF values will be entered into the field logbook so data transcription errors can be discerned easily upon validation. Temperature, pH, specific conductance and turbidity measurements will be transcribed directly from direct read instruments. The information will be entered into the field logbook and checked for transcription errors by the sampling team.

9.1.2 Laboratory Data Reduction Procedures

Reduction procedures in the laboratory will be performed by computer database that will provide printouts of raw data and chromatograms. The information will be evaluated by the bench analyst to ensure proper integration and assignment of various sample constituents. Lab records will note all other information not processed by computer such as reagents, sample preparations, etc.

The department supervisor will review the lab notebook and associated computer printouts to ensure all information is accurate and no errors have occurred. Prior to laboratory release of the data, QA/QC will be performed to assess precision and accuracy requirements of the data have been met.

9.2 DATA VALIDATION

Technical data, including field data and results of laboratory sample analyses, will be validated to monitor the performance of the remedial action. The data collection and quality assurance procedures for validating field and laboratory data are described below.

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 investigative analytical samples.

9.2.1 Procedures Used to Validate Field Data

Validation of data obtained from field measurements will be performed by the ENTACT QA Manager. Such validation will be performed by regularly checking procedures utilized in the field and comparing the data to previous measurements. Data that cannot be validated will also be documented.

Field data requiring validation includes the raw data and supportive documentation generated from field investigations and will include, but is not limited to, the following:

- Field notebooks
- Field investigation daily reports
- Field instrument readings and calibration data sheet;
- Field log borings;
- Sample labels;
- Chain-of-custody forms;
- Sample tracking records;
- Surveying information; and
- Maps.

Field measurements that could affect the quality of the data (such as temperature, pH, conductivity, and water level) will also be validated. Validation of all field data will be performed in terms of meeting DQOs by checking the procedures utilized in the field and comparing the data to previous measurements. The following areas will be addressed during validation:

- Sampling methodology;
- Sample holding times and preservation;
- Field instrument selection and use;
- Field instrument calibration and standardization;
- Field instrument preventative and remedial maintenance;

- Field deviations; and
- Units of measure and reference points from which field data will be measured.

Additional specific evaluations of data critical to the integrity of the decision making process for this task will be performed on 10 percent of the data and will include:

- Chain-of-custody integrity check;
- Review of the appropriateness of field methodologies;
- Transcription, calculation, completeness, and accuracy check of field data; and
- Analysis of field notes to determine presence of bias.

If substantial errors are detected which impact data quality, the scope of the validation will be increased to determine the extent of the problems.

9.2.2 Procedures Used to Validate Lab Data

Under the direction of the Laboratory QA Manager, lab data will be reviewed to ensure that results for samples meet all method-specified criteria. The requirements to be checked in validation are:

- Sample Holding Times
- Calibration
- Blanks
- Matrix Spike/Matrix Spike Duplicate
- Field Duplicate
- Target Compound Identification
- Spectral Interference Check Sample Analysis
- Compound Quantitation and Reported Detection Limits
- System Performance
- Overall Assessment of Data
- Interference Check Sample Analysis
- Laboratory Control Sample Analysis

One equipment blank will be prepared and documented for every 10 investigative samples to assess the accuracy of sampling techniques. One matrix spike and matrix spike duplicate will be analyzed for every 20 investigative samples.

The laboratory QA Manager will be responsible for assessing data quality and advising appropriate laboratory section supervisors of any data that are "unacceptable" or have notations that would caution the data user to possible unreliability. Data reduction, validation, and reporting by the laboratory will be conducted as follows:

- Raw data produced by the analyst will be turned over to the respective supervisor.
- The supervisor will review the data for attainment of QC criteria as outlined in method protocols and established U.S. EPA methods.
- Upon completion of analytical testing, the laboratory project manager conducts a final review.
- Upon acceptance of the data by the laboratory project manager, a computerized report will be generated and sent to the ENTACT QA Manager.
- The ENTACT QA Manager will complete a thorough audit of all reports.

The ENTACT QA Manager will conduct an evaluation of data reduction and reporting by the laboratory. These evaluations will consider the finished data sheets, calculation sheets, document control forms, blank data, duplicate

data, and recovery data for matrix and surrogate spikes. The material will be checked for legibility, completeness, and the presence of necessary dates, initials, and signatures. The results of these checks will be assessed and reported, noting any discrepancies and their effect upon acceptability of the data. In addition, the QA Manager will check for data consistency by assessing comparability of duplicate analyses, comparability to previous criteria, transmittal errors, and anomalously high or low parameter values. The results of these checks will be reported in writing.

The following is a description of the validation steps that will be used by the ENTACT QA Manager to validate the laboratory data. These validation results will be summarized in the Final Report. The validation steps are as follows:

- Compile a list of all samples
- Compile a list of all QC samples
- Review laboratory analytical procedures and instrument performance criteria
- Specific evaluations critical to the integrity of the data include:
- Review of chain-of-custody documents for completeness and correctness;
- Transcription, calculation, completeness, and accuracy check; and
- Review of laboratory analytical procedures, appropriateness, and instrument performance criteria.

In addition, data validation will be performed on 10 % of the confirmational soil and treatment verification data, as consistent with approved U.S. EPA protocol at previous Superfund projectes conducted by ENTACT in Ohio. If significant errors that affect data quality are detected, the percentage of raw data validated will be increased to assess the magnitude of the problem.

- A data summary will be prepared and will include:
  - Results;
  - Sample media identification
  - Sample location and description;
  - Appropriate concentration units;
  - Appropriate significant figures;
  - Data qualifiers; and
  - Definitions

  - The laboratory data summary will be reviewed for potential data quality problems, including:
    - Unexpected results;
    - Common laboratory contaminants;
    - Samples in which dilution was necessary;
    - Time and date of sample collection.

A sample data summary will be prepared to assess precision, accuracy, and completeness of the analytical data. Laboratory records and data package requirements will be checked to assess completeness of the data package. The validation effort will be done by personnel qualified and experienced in the field of laboratory data validation.

Despite all efforts to achieve the objectives of the project, the potential for error exists in laboratory chemical analyses and in the data reporting process. Every reasonable effort will be made to compare and double-check data reported from the laboratory with data entered into the data base management system.

9.3 DATA REPORTING

Data generated during the MMI removal activities will be appropriately identified, validated, and summarized in monthly progress reports, and included in the final report. The ENTACT QA Manager will develop a data storage and information system to facilitate and manipulate data for tracking, data calculations, and transfer of data to various forms and reports and transmittal of data into a data storage system. Data packages from the laboratory will be in the form of a Level 3 QC package excluding a sample traffic report and electronic deliverables.

Data reporting to the ENTACT QA Manager will be performed by the ENTACT QA Technician and the Field Coordinator. After data validation and reduction, the ENTACT QA Technician will report data to the ENTACT QA Manager. The ENTACT QA Manager will summarize the data obtained and include the information in the field activity report submitted to the Project Manager for review. The ENTACT Project Manager will then prepare monthly reports and the final report to the U.S. EPA Project Coordinator. The appropriate documents will be prepared and distributed that summarize both the field activities performed and the results obtained. The field reports will include: presentation of results, summaries of field data from field measurements, and field location of sampling points. All other information will be bound in the appendices. The laboratory reports will include at a minimum the following components:

- Report title page;
- Date of issuance;
- Any deviations from the intended analytical strategy;
- Laboratory batch number;
- Number of samples and respective matrices;
- Project name and number;
- Condition of samples;
- Discussion of holding times;
- Discussion of technical problems or observations;
- Discussion of quality control checks which failed;
- Sample description information;
- Analytical tests assigned;
- Analytical results;
- Quality control reports;
- Description of analytical methodology;
- Description of QC methodology; and
- Signature of Laboratory Operations Manager.

Both the field and laboratory reports will contain the following:

- Any changes in the QA Project Plan;
- Significant QA problems, recommended solutions, and results of corrective actions;
- Discussions of whether the QA objectives were met, and the resulting impact on decision making; and
- Limitations on the use of the measurement data

10.0 PERFORMANCE AND SYSTEMS AUDITS

Two types of audit procedures will be used to assess and document performance and project staff: system audits and performance audits. These audits are performed at frequent intervals under the direction of the ENTACT QA Manager to evaluate quantitatively the accuracy of the total measurement system. These audits form the basis for corrective action requirements and provide a permanent record of the conformance of measurement systems to QA requirements.

System audits consist of quantitative evaluation of field and laboratory quality control measurement systems to determine if they are used appropriately. These audits may be carried out before all systems are operational, during the program, or after the completion of the program. These audits involve a comparison of the activities presented in the QA plan with those actually scheduled or performed.

Performance audits are a quantitative evaluation of the measurement systems of the program. They require testing of the measurement systems with samples of known composition or behavior to evaluate precision and accuracy after systems are operational and generating data. Analytical laboratories designated to perform analytical services during the removal action at MMI will be audited prior to sample analysis.

10.1 INTERNAL AUDITS

A systems audit will be performed prior to or shortly after systems are operational on laboratory, office, and field operations. The system audit protocols are summarized as follows:

Laboratory Operations: Laboratory QA Manager
- Parameter and/or laboratory notebooks;
- Instrument/equipment logbook;
- Sample log-in, routing, and labeling for analysis; and
- Updating of QC criteria for spike recoveries. In addition, the QA Manager will monitor analyses to assure complete adherence to approved analytical methods.

Field Operations: ENTACT QA Officer
- Field notebooks, procedures, field logs, boring logs, etc.
- Site safety;
- Sampling methods; and
- Sample labeling, packing, storage, shipping, and chain-of-custody procedures.

Office Operations: ENTACT Administrative Project Manager
- Project team members are informed of the team organization and in particular the quality control procedures for their work assignment; and
- Quality control officers assigned to the project are available and informed of the quality control they are responsible for, and the schedule for quality control review.

After systems are operational and generating data, a performance audit will be conducted at least once during the laboratory, office, and field work to determine the accuracy of the total measurement systems or component parts thereof. The performance audit protocol is summarized as follows:

Laboratory Operations: Laboratory QA Manager
- Sample log-in, routing, and labeling for analysis;
- Analyses to assure complete adherence to approved test methods; and
- Other quality control procedures outlined herein.

Field Operations: ENTACT QA Officer

- Field notebooks, procedures, field logs, boring logs, etc.
- Site safety;
- Sampling methods; and
- Sample labeling, packing, storage, shipping, and chain-of-custody procedures.

Office Operations: ENTACT Administrative Project Manager

- Specified quality control reviews of the work are being performed;
- The individuals performing the quality control reviews are qualified and as assigned; and
- Final reports and deliverables have received the appropriate QC review.

The auditor will maintain a record of his evaluation by writing field notes. Following the audit, the preliminary results will be reviewed with the person in charge of the operations audited. Subsequent to the audit, the auditor will develop an audit report that summarizes the areas requiring corrective measures. This report will be submitted to the ENTACT Project Manager.

When it is necessary to determine the capacity of a subcontractor's quality assurance program prior to award of subcontractor, the ENTACT Project Manager, ENTACT QA Technician, and/or ENTACT QA Manager will visit the subcontractor's operations to verify performance and services.

10.2 EXTERNAL AUDITS

In addition to these internal field and laboratory audits, the USEPA Region 5 QA reviewer from FSS may conduct external field and laboratory audits. External field and laboratory audits may also be performed by the US EPA Project Coordinator. The external field audits may be conducted any time during the field operations and may or may not be announced. An external audit may be performed at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced. The external lab audit will include (but not be limited to) review of laboratory procedures, laboratory on-site audits, and/or submission of performance verification samples to the laboratory for analysis.

11.0 PREVENTATIVE MAINTENANCE

To minimize the occurrence of instrument failure and other system malfunction, a preventative maintenance program for field and laboratory instruments will be implemented. Equipment, instruments, tools, gauges, and other items requiring preventative maintenance will be serviced in accordance with the manufacturer's specified recommendations and written procedures developed by the operators. Maintenance items that cannot be performed by the laboratory technician will be performed by a person certified to repair the instrument. The laboratory will be responsible for performing routine maintenance and will have available tools and spare parts to conduct routine maintenance. A backup XRF unit will be available for use in the case of a malfunction to avoid downtime.

Manufacturer's procedures identify the schedule for servicing critical items in order to minimize the downtime for the measurement system. It will be the responsibility of the field instrument operator and the laboratory to adhere to this maintenance schedule and arrange any necessary and prompt service. In addition to any manufacturer recommended maintenance criteria, a maintenance procedure will be developed by the operator based upon experience and previous use of the equipment. Service to the equipment, instruments, tools, gauges, etc., shall be performed by qualified personnel. Periodic maintenance is shown on Table QAPP-9.

Logs are used to record maintenance and service procedures and schedules. All maintenance records will be documented and traceable to the specific equipment, instruments, tools, and gauges. Any items found to be inoperable will be taken out of use and a note stating the time and date of this action will be made in the calibration sheets and logs. The reason for equipment failure and the time and date of its return to service will also be noted in the logbook. Records produced shall be reviewed, maintained, and filed by the operators at the laboratories and by the data and sample control personnel when and if equipment, instruments, tools, and gauges are used at the site. The ENTACT Project Manager will audit these procedures.

Table QAPP-9
Maintenance Procedures for Field and Laboratory Equipment

Instrumentation	Maintenance Procedure	Spare Parts
Field XRF	1. Leak testing every six months 2. Shutter check every six months 3. Annual manufacturer servicing	Battery packs XRF Cables
Gas Chromatograph/Mass Spectrometer	1. Change septa as needed 2. Change syringes on autosamplers as needed 3. Leak check when installing columns 4. Injection port cleaning as needed 5. Check inlet system for residue buildup periodically 6. Clean gas line dryers as needed 7. Replace pump oil as needed 8. Replace electron multiplier as needed	Syringe Septa Various electronic components Plumbing supplies Injection port liners
ICP Spectrometer	1. Change sample rinse lines 2. Clean nebulizer components and torch assembly 3. Clean filters 4. Clean mirrors	Nebulizer components Torch assembly Pump tubing Sample probe
Temperature/pH/Conductivity and turbidity meters	1. Calibrate as required by manufacturer's instruction 2. Replace as needed 3. Check batteries if does not calibrate	pH buffers Batteries Spare electrodes

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

This section summarizes the QA/QC procedures used in assessing the quality of the chemical data and the format for presenting the results of the QA/QC evaluations. The data evaluation procedures will be used by the QA Manager for assessing duplicate and spike samples and checking blank samples that are submitted blind to the analytical laboratories from the field or generated internally by the laboratory, in accordance with this QAPP. The purpose of implementing these procedures is to assess the chemical data generated for accuracy, precision, representativeness, and completeness for both the laboratory analytical program and field sample collection activities.

The primary goal of the program is to ensure that the data generated are representative of environmental conditions at the site. Accuracy, precision, representativeness, and completeness will be computed in the manner described in the following paragraphs. A qualitative assessment of accuracy, precision, representativeness, and completeness will be made and documented. The goal of the assessment will be to (1) establish site specific PARCC parameters; (2) use the parameters to develop a database with known limitations of data usability; and (3) evaluate these limitations in achieving the project DQOs. Complex statistical data verification and a significance evaluation will not be performed. If a problem arises and the data are found to deviate from previous analyses or surrounding conditions, the data will be annotated. Sample recollection and analysis will be used only in extreme cases of QC problems.

Chemical data will be evaluated according to accuracy, precision, representativeness, and completeness criteria for both the field sample collection activities and laboratory analytical programs. The QA/QC program will evaluate data based on three types of quality control samples (matrix spikes, blanks, and duplicates).

The completeness of the data represents the amount of valid data obtained from the field programs versus the amount of data expected under normal conditions. Completeness will be assessed prior to preparation of the final report. These procedures for evaluating the field and laboratory QA/QC data are the same and are presented below for QA/QC matrix spike, blank, and duplicate samples.

12.1 ACCURACY ASSESSMENT

In order to assure the accuracy of the analytical procedures, an environmental sample is randomly selected from each sample shipment received at the laboratory, and spiked with a known amount of the analyte to be evaluated. In general, a sample spike should be included in every set of 20 samples tested on each instrument. The spike sample is then analyzed. The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. The percent recovery for a spiked sample is calculated according to the following formula:

$$\% \text{ Recovery} = \frac{\text{Amount in spiked sample - Amount in sample}}{\text{Known amount added}} \times 100$$

12.2 PRECISION ASSESSMENT

Spiked samples are prepared by choosing a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and then spiking each of the aliquots with a known amount of analyte. The duplicate samples are then included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The

relative percent difference (RPD) between the spike and duplicate spike are calculated and plotted. The RPD is calculated according to the following formula:

$$RPD = \frac{\text{Amount in Spike 1 - Amount in Spike 2}}{0.5\ (\text{Amount in Spike 1} + \text{Amount in Spike 2})} \times 100$$

12.3 COMPLETENESS ASSESSMENT

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with a specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{(Number of valid measurements)}}{\text{(Number of measurements planned)}} \times 100$$

13.0 CORRECTIVE ACTION

The following procedures have been established to assure that conditions adverse to quality, such as malfunctions, deficiencies, deviations, and errors, are promptly investigated, documented, evaluated, and corrected. When a significant condition adverse to quality is noted at the site, laboratory, or subcontractor locations, the cause of the condition will be determined and corrective action taken immediately. All project personnel have the responsibility to promptly identify, solicit approved correction, and report conditions adverse to quality. Conditions, which warrant corrective action, include:

- Predetermined acceptance standards are not attained;
- Procedures or data compiled are determined to be faulty;
- Equipment or instrumentation is found to be faulty;
- Samples and test results are questionably traceable;
- Quality assurance requirements have been violated; and
- System and performance audits indicate problems.

13.1 FIELD CORRECTIVE ACTION

The need for corrective action will be identified as a result of the field audits previously described. If problems become apparent that are identified as originating in the field, immediate corrective action will take place. If immediate corrective action does not resolve the problem, appropriate personnel will be assigned to investigate and evaluate the cause of the problem. When a corrective action is implemented, the effectiveness of the action will be verified such that the end result is elimination of the problem.

Corrective action in the field can be needed when the sample network is changed, sampling procedures, and field analytical procedures require modification due to unexpected conditions. In general, the Field Team, Field Coordinator, QA Technician, QA Manager, and Project Manager may identify the need for corrective action. The ENTACT field staff in consultation with the ENTACT Field Coordinator will recommend the corrective action. The ENTACT Field Coordinator will approve the corrective measure, which will be implemented by the ENTACT Field Team. It will be the responsibility of the ENTACT Field Coordinator and the ENTACT Project Manager to ensure that corrective action has been implemented.

If the corrective action will supplement the existing sampling plan using existing and approved procedures in the QAPP, corrective action approved by the ENTACT Field Coordinator will be documented. If corrective actions resulting in fewer samples, alternate locations, etc. which may cause project quality assurance objectives not to be achieved, it will be necessary that all levels of project management, including U.S. EPA, concur with the proposed action.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The ENTACT QA Manager will identify deficiencies and recommended corrective action to the ENTACT Project Manager. Implementation of corrective actions will be performed by the ENTACT Field Coordinator and the ENTACT Field Team. Corrective action will be documented in quality assurance reports to the entire project management. The U.S.EPA will be notified immediately if any problems affecting data quality occur.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by the US EPA Remedial Project Manager.

13.2 LABORATORY CORRECTIVE ACTION

The need for corrective action resulting from QA audits will be initiated by the laboratory QA/QC Manager in consultation with the Laboratory Operations Manager. The corrective action will be performed prior to the release of data from the laboratory. The corrective action will be documented in the logbook and submitted to the data validator. If the corrective action does not rectify the situation, the laboratory will contact the ENTACT Project Manager. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform all levels of ENTACT management at the MMI site and the US EPA Project Coordinator. Corrective action may include, but is not limited to:

- Reanalyzing the samples, if holding time criteria permit;
- Evaluating and amending sampling and analytical procedures;
- Accepting data with an acknowledged level of uncertainty; and
- Resampling and analysis, if the completeness of the data set or intended use of the data is recognized during a preliminary review to be insufficient to meet program DQOs.

If the above corrective actions are deemed unacceptable, an alternate laboratory will be selected to perform necessary analyses.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include resampling by the field team or reinjection/reanalysis of samples by the laboratory. These actions are dependent upon the ability to mobilize the field team, and whether the data to be collected is necessary to meet the required quality assurance objectives (e.g. the holding time has not been exceeded, etc.). The ENTACT QA Manager is responsible for identifying a corrective action situation, documenting the incident, determining the course of action, and implementing the corrective action.

13.4 IMMEDIATE CORRECTIVE ACTION

Any equipment and instrument malfunctions will require immediate corrective actions. The laboratory QC charts are working tools that identify appropriate immediate corrective actions to be taken when a control limit has been exceeded. They provide the framework for uniform actions as part of normal operating procedures. The actions taken should be noted in field or laboratory logbooks. A detailed description of method-specific corrective action limits is provided in the appropriate method. Any deviation from the prescribed control limits must be approved in writing by the ENTACT QA Manager.

13.5 LONG-TERM CORRECTIVE ACTION

The need for long-term corrective action may be identified by standard QC procedures, control charts, and system audits. Any procedural or data quality problem that cannot be solved by immediate corrective action becomes a long-term corrective action. The essential steps in a corrective action system are as follows:

- Identification and definition of the problem;
- Investigation and determination of the cause of the problem;
- Determination and implementation of a corrective action to eliminate the problem; and
- Verification that the corrective action has eliminated the problem.

Documentation of the problem is important in corrective action. The responsible person may be an analyst, ENTACT QA Manager, laboratory QA Manager, sampler, or the ENTACT Project Manager. In general, the designated QA Manager will investigate the situation and determine who will be responsible for implementing the corrective action. The QA Manager will verify that the corrective action has been taken, appears effective, and that the problem has been resolved.

The required corrective action will be documented by the designated ENTACT QA Manager and the ENTACT Project Manager for field activities. The corrective action will be discussed with the ENTACT Project Manager and the EPA Project Manager prior to implementation if the severity of the problem warrants such discussion.

Any changes proposed for amending sampling and analytical procedures will be approved by the EPA prior to implementation. These changes will be documented in monthly progress reports and addenda to the QAPP.

Project management and staff, including field investigation teams, document and sample control personnel, and laboratory groups, will monitor on-going work performance in the normal course of daily responsibilities. Work will be monitored at the site by the ENTACT Project Manager.

Following identification of an adverse condition or quality assurance problem, the ENTACT QA Manager will notify the ENTACT Project Manager of the problem.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.1 CONTENTS OF A PROJECT QA REPORT

Analytical results of samples analyzed during the remedial action will be submitted to the Project Manager following a QA/QC review. The results will include a tabulation of the analytical data and an explanation of any field conditions or laboratory QA/QC problems and their effects on data quality. Results of performance audits and system audits will also be included, as appropriate. Proposed corrective action will be recommended in the event that QA problems are identified during review of data quality or results of performance or system audits.

The final report will contain a discussion of QA/QC evaluations summarizing the quality of the data collected and/or used as appropriate to each activity of the project. The objective of the QA/QC summary will be to ensure that the data are representative of site conditions and sufficient in quality and quantity to support the field activities. The QA/QC summary will include:

- Tabulated results of all field and analytical data;
- A report from the laboratory QA Manager evaluating the validity of the analytical data with respect to accuracy, precision, completeness, and representativeness; and
- A report from the Project Manager evaluating the results of field and office audits.

A quality assurance report will be prepared by the QA Manager upon receipt of sufficient QA data from the laboratory. The report will be a summary of QA/QC results of the analytical work conducted and will be included as part of the final remedial action report.

14.2 QA REPORTING AND ROUTING SCHEDULE

The QA Reports will be prepared on a monthly basis and will be delivered to all recipients by the end of the first full week of the month. The reports will continue without interruption, until the project has been completed. All individuals identified in the Project Organization Chart will receive copies of the monthly QA Report.

ATTACHMENT QAPP-A

PACE ANALYTICAL STANDARD OPERATING PROCEDURES

ATTACHMENT QAPP-B

GEOANALYTICAL, INC. LABORATORY STANDARD OPERATING PROCEDURES

Attachment QAPP-B1

Inductively Coupled Plasma-Mass Spectrometry
Method 6020

Inductively Coupled Plasma-Atomic Emission Spectroscopy
Method 6010A

GAM 6020
Inductively Coupled Plasma-Mass Spectrometry
Revision 3.0: 02/11/02

ANALYTES*:		CAS #
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-43-9
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Mercury	(Hg)	7439-97-6
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Thallium	(Tl)	7440-28-0
Thorium	(Th)	7440-29-1
Tin	(Sn)	7440-31-5
Titanium	(Ti)	7440-32-6
Uranium	(U)	7440-61-1
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

*see table 1 for analyte approval/ method acceptability status

INSTRUMENTATION: ICP-MS

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-µg/L concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in aqueous samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required. Water samples with a turbidity of <1 NTU do not require digestion prior to analysis and may undergo "direct analysis" (applicable to method 200.8 only).

1.2 ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability of Method 6020 rev. 0 in a multi-laboratory study on solid

wastes are listed in table 1. Acceptability for method 6020 rev. 0 for an element was based upon the multi-laboratory performance compared with that of either furnace atomic absorption spectroscopy or inductively

coupled plasma-atomic emission spectroscopy. It should be noted that the multi-laboratory study was conducted in 1986. Multi-laboratory performance data for the listed elements (and others) are provided in Section 9. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, instrumentation, and operating conditions. In relatively simple matrices, detection limits will generally be below 0.02µg/L. Since 1986 when the initial analyte list was established for method 6020 rev. 0, significant improvements in instrumentation have been made. Research has been performed and knowledge gained regarding additional analyte suitability for quantitation using this method. This is illustrated in table 1. It should be noted the proposed update (6020 rev. 1) lists additional analytes as does method 6020 CLP-M ver. 9.0 as well as method 200.8. Additional analytes quantitated under method 6020 rev. 0 are noted in table 1. These analytes have demonstrated to perform to adequate acceptability standards. The analyst is required to monitor potential sources of interferences and take appropriate action to ensure data of known quality (see section 8.0).

1.3 Use of this method is restricted to spectroscopists who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.

1.4 An appropriate internal standard is required for each analyte determined by ICP-MS. Internal standards are prepared at a concentration of 150 ppb and introduced on-line using a second channel of the peristaltic pump (if on-line addition is not used, then internal standard spiking may be performed by adding a constant volume of internal standard concentrate to identical volumes of the standards and prepared samples). See table 2 for internal standards used, 8.2 for internal standard quality control criteria, and 8.3 for internal standard intensity monitoring. See also 5.8 and 7.8.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples which require total ("acid-leachable") values must be digested using appropriate sample preparation methods (e.g. Methods 3005A (GAM3005) for metals in water, Methods 3050B (GAM 3050B) for metals in soil, and 3005A (GAM3005A). When analyzing for dissolved constituents, water samples may be analyzed directly (without digestion) after acid matrix matching provided the sample has been filtered through a 0.45-um filter and preserved with nitric acid to pH < 2. Water samples may also be analyzed directly (without digestion) after matrix matching for methods 200.7 and 200.8 if sample turbidity is < 1 NTU.

2.2 This method describes the multi-elemental determination of elements by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the

plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with an off-axis discrete dynode electron multiplier (DDEM). Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

3.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system is used to correct for these interferences (see table 2). This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal.

3.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified. Examples include ArCl+ ions on the 75AS signal and MoO+ ions on the cadmium isotopes.

Corrected arsenic signal (using natural isotopes abundances for coefficient approximations) =

(m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal), (where the final term adjusts for any selenium contribution at m/z 77).

NOTE: As values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than 82Se+, (e.g. 81BrH+ from bromine wastes).

Similarly,

corrected cadmium signal (using natural isotopes abundances for coefficient approximations)=

(m/z 114 signal) - (0.027)(m/z 118 signal) - (1.63)(m/z 108 signal), (where last 2 terms adjust for and tin or MoO+ contributions at m/z 114).

NOTE: Cadmium values will be biased low by this type of equation when 92ZrO+ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct (94ZrOH+) and indirect (90ZrO+) additive interferences when Zr is present.

3.3 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in

matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. Analyst Note: In the performance of this method, it has been observed that the use of new or newly cleaned cones results in large initial changes in the ion transmission efficiencies. These changes may produce a large instrumental drift which can cause drift-sensitive quality assurance parameters to exceed control limits. It has been found that by conditioning the cones, via exposure to solutions (such as the ICS) which are similar to the samples analyzed, the changes in ion transmission efficiencies will be mitigated. This conditioning appears to form an oxide layer on the cones which insulates and therefore stabilizes the ion transmission efficiencies.

3.4 Memory interferences can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

3.5 Abundance sensitivity - Is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences must be recognized and the spectrometer resolution optimized to minimize them.

Many commercial instruments provide unit resolution at 10% peak height. At this peak height very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. UltraMass 700 provides unit resolution at 5% peak height. This type of interference is uncommon but is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled argon plasma-mass spectrometer:(Varian UltraMass 700 Serial #96072018 (instrument #37).

Mass Analyzer: quadrupole
Resolution: < 1 amu at 5% peak height
Mass Range: 3 to 256 amu
Quadrupole Assembly:
rod diameter: 9.5 mm
rod length: 200 mm
Interface

sampler: Ni- 1.0 mm orifice
skimmer: Ni- 0.5 mm orifice
Spray Chamber: Sturman-Masters- double pass cyclonic
Nebulizer: Meinhard glass concentric (style C or K)
Detector: Discrete dynode electron multiplier (off-axis)
System software capable of corrections for isobaric interferences and application of the internal standard technique. A mass-flow controller and four channel variable-speed peristaltic pump are also used.

4.1.1 Argon gas supply (AGA 45 or 52 gallon liquid).

4.2 Class A volumetric flasks: 5-, 10-, 25-, 50-, and 100-mL.

4.3 Class A TD volumetric pipettes: 0.5-, 1.0-, 2.0-, 3.0-, 4.0-, 5.0-, and 10-mL.

4.4 MLA Precision air displacement pipetters: 10-, 25-, 50-, 100-, 250-, and 1000-uL. (or equivalent)

4.5 Analytical balance- capable of accurate measurement to 0.01g.
(Sartorious PT 120 ID#30121328 (120g max))
(Mettler-Toledo PB602 (610g max))
Analytical balance- capable of accurate measurement to 0.0001g.
(Sartorious A200S)

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.(e.g.Mallinckrodt from VWR).

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an SMI TopSider Series 2 liquid dispenser.

5.1.3 Nitric acid (conc), HNO3.(e.g.Tracepur Plus by EM from VWR).

5.1.4 Nitric acid (1:1), HNO3. Add 500 mL concentrated HNO3 to 400 mL water and dilute to 1 liter in an SMI Topsider Series 2 liquid dispenser.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water must meet ASTM type I standards (18 mΩ or greater). Reagent water will be interference free. Reagent water is dispensed from a Barnstead Nano-Pure unit Model #D4741, Serial #687920145027. Cartridges are changed approximately every six months. The Barnstead unit is fed water from a Kinetico commercial

reverse osmosis unit (serial #361001). This unit has the capability to produce 75 gallons of water per day and has a 20 gallon bladder tank for storage.

5.3 Standard stock solutions may be purchased from a number of different manufacturers. The manufacturer used to prepare ICP-MS standards is Inorganic Ventures. Table 4 gives a list of common single-element and custom blend multi-elemental standard concentrations and common dilution concentrations used for calibration and calibration verification purposes.

5.4 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.4.1 The calibration blank consists of the same concentration of the same acid used to prepare the final dilution of the calibrating solutions of the analytes (usually 1 or 2% HNO_3 (v/v) in reagent water) along with the selected concentrations of internal standards such that there is an appropriate internal standard element for each of the analytes (see table 2 for internal standards used). This sample is analyzed prior to an analytical sequence run, after every 10 samples, and at the end of an analytical run (see section 8.7 for use).

5.4.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions (see section 8.7 for use).

5.4.3 The rinse blank consists of 1 or 2% percent HNO_3 (v/v) in reagent water. Add 200 ug/L Au for mercury determination.

5.5 The interference check solutions are prepared by diluting multi element interferent check standards purchased from Inorganic Ventures. The first standard contains known concentrations of interfering elements that will provide an adequate test of the correction factors. The second standard contains elements of interest at low concentration. Table 5 contains a list of the elements within the interferent check standards and their concentrations along with the diluted concentrations. Within an analytical run standard #1 must be diluted and analyzed by itself (contains interfering elements) and immediately following standards #1+#2 must be diluted, mixed together and analyzed (see step 8.7.2). The interferent check standards are run after initial calibration verification or once every 12 hours, whichever is more frequent.

NOTE: Interferent check solutions not required for method 200.8

5.6 The quality control standard is the initial calibration verification

solution (ICV), which must be prepared in the same acid matrix as the calibration standards (see table 4). This solution is an independent standard. An independent standard is defined as a standard composed of the analytes from a source different from those used in the standards for instrument calibration (second source).

5.7 Mass spectrometer tuning solution (see table 6). A solution containing elements representing all of the mass regions of interest must be prepared to verify that the resolution and mass calibration of the instrument are within the required specifications. This solution is also used to verify that the instrument has reached thermal stability and to check the quadropole resolution (see 7.5, 7.6).

5.8 Internal standards. Internal standards are used to correct for short term instrument drift, physical interferences (see 8.2) and monitoring long term instrument drift (as reduction of intensity) (see 8.3). Five or more analytes are monitored as internal standards that effectively cover the entire mass range (see table 2). Internal standards must be present in all samples, standards and blanks at identical levels. This is achieved through on-line addition (see 1.4). The on-line addition method preserves the concentration of the internal standard solution when dilutions are necessary.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous samples shall be preserved to pH <2 with nitric acid and have a holding time of 6 months for analysis of all metals except mercury which has a holding time of 28 days. Sample preservation is performed by the sampler immediately upon sample collection. Samples are filtered immediately on-site by the sampler before adding preservative for dissolved elements.

Soil samples have a holding time of 6 months for all metals except mercury which has a holding time of 28 days. The preservation required for soil samples is maintenance at 4°C (+/- 2°C) until digestion.

NOTE: Holding times are calculated from the date when the sample was collected.

7.0 PROCEDURE

7.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Solubilization and digestion procedures are presented in sample preparation methods 3005A(GAM3005A), 3010(GAM3010), 3050B(GAM3050B), 1311(GAM1311), 1312(GAM1312).

7.2 Due to the sensitivity and intolerance for dissolved solids in the ICP-MS technique, soil samples receive an initial dilution (usually 1:20 or 1:10) and aqueous samples receive an initial dilution (usually 1:10 or 1:5). However, for dissolved constituent analysis or direct analysis aqueous samples may not require digestion. Dilutions are commonly

performed using the autodiluter accessory. See appendix A for use of the autodiluter diluter accessory.
NOTE: If Mercury is to be determined, 200ug/L Au is added to all blanks, standards, and samples.

7.3 Review/follow the daily setup and maintenance procedures in table 8.

7.4 The instrument must be allowed to become thermally stable before beginning (requiring 30 minutes of warm-up prior to calibration). The gate valve must be open during the warm-up period in order to stabilize the temperature of the quadruple and ion optics.

7.5 Perform a mass calibration using tuning solution (see 5.7). If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value (see table 7). Print results of mass calibration and place in logbook.

7.6 Immediately after the mass calibration is performed conduct a performance test using tuning solution (see 5.7) to verify instrument stability, resolution and performance. Re-perform performance test or make adjustments and reperform performance test if criteria in table 7 are not met. Print results of performance test and place in logbook.

NOTE: The performance test also verifies resolution which must meet criteria as specified in table 7.

7.7 If the ion optics/ quadrupole assembly has been taken out, the quadruple resolution must be reset. The resolution must be less than 0.9 amu full width at 5% peak height. Optimum resolution is .76 amu for the low mass range and .74 amu for the high mass range. If the quadruple resolution is reset print results and place in logbook.

7.8 At this point verify that the internal standard is being introduced on-line (see 1.4, 5.8, 8.2 and 8.3).

7.9 Calibrate the instrument for the analytes of interest using the calibration blank (see 5.4.1) and at least a single initial calibration standard (see 5.3). The calibration blank is presented first followed by the standard(s) in order of increasing concentration. Flush the system with the rinse blank (see 5.4.3) between each solution. Use the average intensity of three exposures for both standardization and sample analysis.

7.10 All masses which could affect data quality are monitored to determine potential effects from matrix components on the analyte peaks.

7.11 Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of the calibration verification solution (see 5.6). When measurements exceed + or - 10% of the accepted value, the analyses must be terminated, the problem corrected, the instrument recalibrated, and the new calibration verified. Any samples analyzed under an out-of-control calibration must be reanalyzed.

NOTE: During the course of an analytical run, the instrument may be "resloped" or recalibrated to correct for instrument drift. A recalibration must then be followed immediately by a new analysis of a CCV and CCB before any further samples may be analyzed.

7.12 Flush the system with the rinse blank solution (see 5.4.3) for at least 30 seconds before the analysis of each sample, standard, blank, or check solution. Nebulize each sample until a steady-state signal is achieved (15 - 30 seconds) prior to collecting data. Analyze the calibration verification solution (see 5.6) and the calibration blank (see 5.4.1) at a frequency of once every 10 analytical samples. The calibration verification solution must be within + or - 10% of the actual value (see 7.11). The calibration blank must meet QC requirements as specified in 8.7.1.2.

7.13 Samples that are more concentrated than the linear range are automatically diluted and reanalyzed using the auto-diluter.

7.14 Calculations: The quantitative values are reported in units of micrograms per liter (µg/L) for aqueous samples and milligrams per kilogram (mg/kg) for solid samples. If dilutions were performed, the appropriate corrections must be applied to the sample values. Solid samples may be reported on a dry-weight basis. Calculations include appropriate interference corrections, internal-standard normalization, and the summation of signals at m/z 206, 207, and 208 for lead (to compensate for any differences in the abundance of these isotopes between samples and standards). See table 14 for calculation equations.

8.0 QUALITY CONTROL

8.1 Detector scans are performed approximately every three months. Detector voltage is optimized if necessary. Detector should be replaced once optimized voltage reaches 2700-3000V. New detectors will have initial optimized voltage between 1600-1900V.

8.2 The intensities of all internal standards must be monitored for every analysis.

For method 6020: When the intensity of any internal standard fails to fall between 30 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4)(1:5) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal standard intensities fall within the prescribed window.

For method 200.8: The absolute response of any one internal standard must not deviate more than 60-125% of the original response in the

calibration blank. If deviations greater than these are observed, flush the instrument with the rinse blank and monitor the responses in the calibration blank. If the responses of the internal standards are now within the limit, take a fresh aliquot of the sample, dilute by a further factor, add the internal standards and reanalyze. If after flushing the response of the internal standards in the calibration blank are out of limits, terminate the analysis and determine the cause of the drift.

8.3 When the ongoing daily internal standard intensity monitoring shows original intensities at a 30- 50% reduction of the original reading, the sampler and skimmer cones should be cleaned.

8.4 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent. See table 9 for spiking concentrations.

8.4.1 Calculate the values for the preparation blanks, laboratory control samples (LCS), matrix spikes(MS) and matrix spike duplicates(MSD)

8.4.1.1 If the preparation blank shows contamination at a level of less than the (MRL) method reporting limit, less than 5% of the regulated limit for that element or less than 5% of the concentration of the analyte present in sample, the method is presumed in control and sample analysis can proceed. If contamination is detected above the reporting limit it must be included in the analytical report.

8.4.1.2 If the preparation blank contains contamination above the reporting limit, and greater than 10% of the concentration of the analyte of interest present in the samples associated with the blank, corrective actions must be performed to bring the method back into control. After the corrective actions are performed the analyst(s) must demonstrate that the preparation and analysis procedures are free of contaminants before sample analysis can proceed. See 8.8.1.

8.4.1.3 Calculate the spike recoveries for the LCS, MS and MSD. If all recoveries are within the established limits in table 10 the method is presumed in control and sample analysis can proceed.

8.4.1.4 If the spike recoveries for the LCS are within the established control limits in table 10, but the MS (and/or MSD) are not within the established limits in table 10, the method is presumed in control and sample analysis can proceed. Sample data for the spiked sample with recoveries outside of the acceptance limits in table 10 should be flagged as "estimated concentration."

8.4.1.5 If the spike recoveries for the LCS are not within the established control limits in table 10, corrective actions must be performed to bring the method back into control. After corrective actions are performed, the analyst(s) must demonstrate LCS recoveries within the established limits before sample analysis can proceed. See 8.8.2.

8.5 Samples that are more concentrated than the linear range are auto-diluted and reanalyzed.

8.6 Two additional tests are performed as required. These tests, as outlined in Steps 8.6.1 and 8.6.2, will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

8.6.1 Serial dilution: If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally, a factor of at least 100 times the instrument detection limit), an analysis of a fivefold (1+4) (1:5) dilution must agree within +/- 10% of the original determination. If not, the sample should be suspect of an interference effect. One serial dilution must be analyzed for each analytical batch or 20 samples whichever is more frequent. It is common practice, but not necessary to do a serial dilution on the same sample that a matrix spike and duplicate matrix spike were performed on.

8.6.2 Post-Digestion Matrix spike addition: To gain additional information on the characteristics of a sample, a post-matrix spike addition may be performed. An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125 percent of the known value. The spike addition should be based on the actual or suspected indigenous concentration of each element of interest in the sample. If the spike is not recovered within the specified limits, additional dilutions may be performed. It is common, but not necessary to do a post-digestion matrix spike addition on the same sample that a serial dilution and matrix spike and duplicate matrix spike were performed on.

8.7 Check the instrument standardization by analyzing appropriate check standards as follows.

8.7.1 Verify calibration every 10 samples and at the end of the analytical run, using a calibration blank (a.k.a.:ICB,CCB Step 5.4.1) and a check standard (a.k.a.:ICV,CCV Step 5.6, 7.11) See table 11 for typical run sequence.

8.7.1.1 The results of the check standard are to agree within 10% of the expected value; if not, rerun the instrument check standard. If it is not within 10% for a second time terminate the analysis, correct the problem, and reanalyze the samples associated with that check standard. (A reslope or

recalibration may be performed see note to 7.11).

8.7.1.2 The results of the continuing calibration blank are not to exceed three times the Instrument Detection Limit (3 X IDL).

8.7.2 Analyze the interference check sample (Step 5.5) at the beginning of an analytical run (immediately after calibration verification (ICV, ICB)) or once every 12 hours, whichever is more frequent. This verifies the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections. The results of the A interference check solution must not exceed the element reporting limit. The results of the AB interference check solution must not exceed the true value +/- 20% or +/- the reporting limit from the true value, whichever is greater.

NOTE: Analytical values are not required for the analytes present in ICS solution A.

NOTE: Interferent check solutions not required for method 200.8

8.8 Corrective Actions

8.8.1 If the preparation blank is out of control (see 8.4.1.2) the following procedures are required.

8.8.1.1 Check to be sure there are no erros in calculation. Also, check instrument performance.

8.8.1.2 Recalculate the data and/or re analyze the sample if any of the above checks reveal a problem.

8.8.1.3 Re-analyze or re-digest and re-analyze the prep blank to demonstrate that the analysis is in control.

8.8.1.4 Re-digest and re-analyze all samples and quality control samples associated with the unacceptable prep blank.

8.8.2 If the laboratory control sample (LCS) is out of control (see 8.4.1.5) the following procedures are required.

8.8.2.1 Check to be sure there are no errors in calculations, spiking solutions. Also, check instrument performance.

8.8.2.2 Recalculate the data and/or re analyze the sample if any of the above checks reveal a problem.

8.8.2.3 Re-analyze or re-digest and re-analyze the LCS to demonstrate that the analysis is in control.

8.8.2.4 Re-digest and re-analyze all samples and quality control samples associated with the unacceptable LCS.

8.8.3 Flag data from samples that have unacceptable pre-digestion matrix spike recoveries as estimated concentration.

9.0 METHOD PERFORMANCE

9.1 In an EPA multi-laboratory study, 10 laboratories applied the ICP-MS technique to both aqueous and solid samples. Table 12 summarizes the method performance data for aqueous samples. Performance data for solid samples is provided in Table 13.

10.0 REFERENCES

1. U.S. EPA Methods 6020, 3015, 3051, 3005A, 3010A, 3020A, and 3050A. Test Methods for evaluating solid waste, physical/chemical methods, SW-846, 3rd ed., and updates. Washington, DC: Government Printing Office, 1993.
2. Method 200.8. Determination of trace elements in waters and wastes by inductively coupled plasma- mass spectrometry. Methods for the determination of metals in environmental samples, suppl I, U.S.EPA/600/R-94/111. Arlington, VA: National Technical Information Service, 1994.
3. Method 6020 CLP-M version 9.0. Inductively coupled plasma-mass spectrometry CLP-M modified for the Contract Laboratory Program.
4. Wolf R, Denoyer E, Sadowski C, Grosser Z. RCRA SW-846 Method 6020 for the ICP-MS analysis of soils and sediments. Application note ENVA-301. Norwalk, CT: The Perkin-Elmer corp., 1996.
5. Wolf R, Denoyer E, Grosser Z. EPA Method 200.8 for the analysis of drinking waters. Application note ENVA-300A. Norwalk, CT: The Perkin-Elmer corp., 1996.
6. Thomas, George P., American Environmental Laboratory. Pages 28-30, March 1997.

TABLE 1. ANALYTE APPROVAL/ METHOD ACCEPTABILITY STATUS

ANALYTE	Method 6020 rev 0 Sept. 1994	Method 6020A rev 1 Jan. 1998+	Method 6020 CLP-M version 9.0*	VAP certif. Method 6020**	Method 200.8 rev 5.4 May 1994	VAP certif. Method 200.8**	Add. Quantifiable analytes	
Aluminum	X	X	X	X	X	X		
Antimony	X	X	X	X	X	X		
Arsenic	X	X	X	X	X	X		
Barium	X	X	X	X	X	X		
Beryllium	X	X	X	X	X	X		
Cadmium	X	X	X	X	X	X		
Calcium		X	X					
Chromium	X	X	X	X	X	X		
Cobalt	X	X	X	X	X	X		
Copper	X	X	X	X	X	X		
Iron		X	X	X				
Lead	X	X	X	X	X	X		
Magnesium		X	X					
Manganese	X	X	X	X	X	X		
Molybdenum			X	X	X	X		
Mercury		X						
Nickel	X	X	X	X	X	X		
Potassium		X	X					
Selenium		X	X	X	X	X		
Silver	X	X	X	X	X	X		
Sodium		X	X					
Thallium	X	X	X	X	X	X		
Thorium					X			
Tin							X	
Titanium							X	
Uranium					X			
Vanadium		X	X	X	X	X		
Zinc	X	X	X	X	X	X		

\+ Proposed method.
* Information listed for comparative purposes.
** Certification applied for.

TABLE 2. PRIMARY ISOTOPES, SECONDARY ISOTOPES, AND ELEMENTAL EQUATIONS FOR DATA CALCULATION

ELEMENT	ISOTOPE	I/S++	CORRECTIVE EQUATION	NOTE
Al	27	Sc		
Sb	121	In		
Sb	123*	In	- 0.1286 * Te125	
As	75	Y	- 3.1278 * M77 + 1.0177 * Se78	
Ba	135+	Tb		
Ba	137+	Tb		
Be	9	Li		
Cd	111*	In		
Cd	114	In	-0.0149 * Sn118 - 1.6285 * M108	
Ca	44	Sc		
Cr	52	Sc		
Co	59	Sc		
Cu	63*	Sc		
Cu	65	Sc		
Fe	54*	Sc	- 0.02817 * Cr52	
Fe	57	Sc		
Pb	206,207,208*	Bi		
Mg	24	Sc		
Mn	55	Sc		
Mo	95*	In		
Hg	202	Bi		
Mo	98	In	- 0.111 * Ru101	
Ni	60	Sc		
Ni	62*	Sc		
K	39	Sc		
Se	77*	Y		
Se	78	Y	- 0.03043 * Kr83 - 0.1869 * M76	
Se	82*	Y	- 1.009 * Kr83	
Ag	107	In		
Ag	109*	In		
Na	23	Sc		
Tl	205	Bi		
Th	232	Bi		
Sn	118	In		
Ti	47*	Sc		
Ti	49	Sc		
U	238	Bi		
V	51	Sc	- 3.1081 * M53 + 0.3524 * Cr52	
Zn	66	Y		
Zn	68*	Y		

I/S	ISOTOPE	ELEMENTAL EQUATION	NOTE
Li	6	- 0.0813 * Li7	enriched Li6 used to minimize int from native Li
Sc	45		
Y	89		
In	115	-0.0149 * Sn 118	
Tb	159		
Bi	209		

notes: *non-primary isotope
+ 135 commonly used for soil matrix, 137 commonly used for aqueous matrix
• multiple masses are used in calculation of Pb values (206, 207,208) to allow for isotopic variability of Pb isotopes
++ analyte internal standard may vary at operator discretion.

TABLE 3. OPERATION CONDITION PARAMATERS

Conditions	Settings
-------Plasma Gas Flow	14.0-18.0 L/min.
Auxiliary Gas Flow	1.0-1-4 L/min.
Peristaltic Pump Speed	15-20 rpm
Rinse Time	30-60 sec.
Sample Uptake Time	20-45 sec.
Stabilization Time	15-30 sec.
Sampler/Skimmer Cones	Nickel
Scanning Mode	Peak Hopping
Dwell time	750-5000µsec, 10k-100kµsec.*
Number of Scans/Replicate	50-100
Replicate Time	non-editable (calculated based on parameters defined)
Number of Points/Peak	One or three points per peak
Number of Replicates	3-5 replicates

*all elements have a 750-5000µsec dwell time except As, Se and Hg which have a 10k-100kµsec dwell time

TABLE 4. SINGLE ELEMENT AND MULTIELEMENTAL MIXED CALIBRATION STANDARDS

VAR-CAL-1	Sb, Mo, Sn, Ti
VAR-CAL-2	Al, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, Th, U, V, Zn
VAR-MAJOR-1	Ca, Fe, Mg, K, Na
Titanium	Ti
Molybdenum	Mo
Tin	Sn
Antimony	Sb
Mercury	Hg
VAR-QC-2	Al, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, Th, U, V, Zn
VAR-MAJOR-QC1	Ca, Fe, Mg, K, Na

Mixed standards have a concentration of 100 µg/mL (VAR-CAL-1, VAR-CAL-2, VAR-QC-2), and 500 µg/mL (VAR-MAJOR-1, VAR-MAJOR-QC1). Single elemental standards Ti, Mo, and Sn have a concentration of 1000 µg/mL while Sb has a concentration of 100 µg/mL.

From these stock standards, a working standard is prepared as 1mL of VAR-CAL-1, 1mL of VAR-CAL-2, and 1mL of VAR-MAJOR-1 made to 100mL with d.i. acidified with HN03 to appropriate concentration (usually 1 or 2%). From this working standard, calibration standards are prepared as 1mL working into 100mL acidified with HN03 to appropriate concentration (usually 1 or 2%) yielding a 10/50 ppb solution, 5mL working into 100mL acidified with HN03 to appropriate concentration (usually 1 or 2%) yielding 50/250 ppb solution, 10mL working into 100mL acidified with HNO3 to appropriate concentration (usually 1 or 2%) yielding 100/500ppb solution. Differing solution volumes and concentrations may be used as needed.

Quality control standards are prepared using VAR-QC-2 and VAR-MAJOR-QC1 and single-element standards. Concentrations and volumes may vary as necessary.

Fresh multi-element calibration standards are prepared every two weeks or as needed and recorded in the ICP-MS working standards logbook.

Additional single-element standards may be used and made up at appropriate concentrations as needed.

TABLE 5. RECOMMENDED INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS.

Element	INT-A(6020ICS-OA) stock concentrations	INT-B(6020ICS-OB) stock concentrations	*ICS-A diluted concentrations	**ICS-A + ICS-B diluted concentrations
Al	1000 ug/mL		10 mg/L	10 mg/L
Ca	1000 ug/mL		10 mg/L	10 mg/L
Fe	1000 ug/mL		10 mg/L	10 mg/L
Mg	1000 ug/mL		10 mg/L	10 mg/L
Na	1000 ug/mL		10 mg/L	10 mg/L
P	1000 ug/mL		10 mg/L	10 mg/L
K	1000 ug/mL		10 mg/L	10 mg/L
S	1000 ug/mL		10 mg/L	10 mg/L
C	2000 ug/mL		20 mg/L	20 mg/L
Cl	10000 ug/mL		100 mg/L	100 mg/L
Mo	20 ug/mL		0.2 mg/L	0.2 mg/L
Ti	20 ug/mL		0.2 mg/L	0.2 mg/L
As		2 ug/mL	0.0 mg/L	0.0200 mg/L
Cd		2 ug/mL	0.0 mg/L	0.0200 mg/L
Cr		2 ug/mL	0.0 mg/L	0.0200 mg/L
Co		2 ug/mL	0.0 mg/L	0.0200 mg/L
Cu		2 ug/mL	0.0 mg/L	0.0200 mg/L
Mn		2 ug/mL	0.0 mg/L	0.0200 mg/L
Ni		2 ug/mL	0.0 mg/L	0.0200 mg/L
Ag		2 ug/mL	0.0 mg/L	0.0200 mg/L
Zn		2 ug/mL	0.0 mg/L	0.0200 mg/L

*these concentrations are obtained by diluting ICS-A 1:100 (1mL ICS-A into 100mL volumetric flask, bring to volume with appropriate acid concentrations and deionized water)

**these concentrations are obtained by diluting ICS-A 1:100 and ICS-B 1:100 (1mL ICS-A + 1mL ICS-B into 100 ml volumetric flask, bring to volume with appropriate acid concentrations and deionized water)

TABLE 6. TUNING SOLUTION

VAR-TS-MS	Ba, Be, Ce, Co, In, Pb, Mg, Tl, Th

note: VAR-TS-MS has a concentration of 10 µg/mL. Tuning solution is prepared at a concentration of 100 µg/L by adding 1 mL VAR-TS-MS : 100 mL d.i. water.

TABLE 7. MASS CALIBRATION AND SYSTEM PERFORMANCE TEST CRITERIA.

MASS CALIBRATION

	EPA 200.8	SW846 6020
Mass Shift Limit	< or = 0.1 amu	< or = 0.1 amu

PERFORMANCE TEST

	EPA 200.8	SW846 6020
Replicates Required	minimum of 5	minimum of 4
Instrument Stability Requirement	RSD < or = 5%	RSD < 5%
Resolution Requirements	approximately 0.75 amu at 5% peak height recommended	<0.9 amu full width at 10% peak height required
Ratio CeO/Ce	< 3% recommended	< 3% recommended
Ratio Ba++/Ba	< 5% recommended	< 5% recommended
Monitor Background at Mass	228 or 220	228 or 220

Note: Mass Calibration and Performance Test use tuning solution with analytes covering the entire
mass range (see table 6).

TABLE 8. DAILY SETUP/SHUTDOWN AND MAINTENANCE PROCEDURES

DAILY SETUP

Note: ICP-MS mains instrument power, PC power, exhaust fans, recirculator and vacuum system
remain on continuously.
Perform any maintenance necessary (see MAINTENANCE section below).
If torch was replaced, set torch at proper position using torch tool.
Attach peristaltic pump tubing (sample, internal standard, and drain).
Light plasma.
Open gate valve by going to plasma alignment page and clicking on time scan.
Allow instrument to become thermally stable by allowing it to warm up for
30 minutes.
Perform a torch alignment if torch was replaced.
Perform a mass calibration (see 7.5).
Conduct a performance test (see 7.6).

MAINTENANCE

Recirculator	Check water level in recirculator (weekly). Vacuum/ and or blow clean with compressed air the heat exchanger fins (as needed).
Spray Chamber flow	Inspect for deposits, dirt, debris that may restrict solution and affect instrument performance (daily). Replace o-rings (as necessary). Clean in ultrasonic bath as needed.
Concentric Nebulizer flow and Tubing	Inspect for deposits, dirt, debris that may restrict solution affect instrument performance (daily). Replace as needed.
Gas Supply	Check Ar level (daily). Maintain a pressure of 90 p.s.i.
Torch and	Inspect for deposits, dirt, debris that may impair measurements effect instrument performance (daily).
Cabinet	Vacuum ventilation fins (as needed). Keep external cabinet clean (daily).
Induction Coil	Inspect coil for signs of deterioration/corrosion.
Vacuum System	Check oil levels on roughing pumps. Top off as necessary. Change oil in roughing pumps when it becomes dirty. Note: the pump oil levels can be viewed while the instrument is operating, however, a more accurate check of the rotary pump oil can be performed when the pump is off. The oil level should be at least half way up the window when the pump is NOT operating. Note: the roughing pump for the plasma sampling interface will need to be changed at a greater frequency than the turbo-backing roughing pump. Turbomolecular pumps are not user serviceable and require no maintenance.
Sample/Skimmer Cones	When the ongoing daily internal standard intensity monitoring shows original intensities at a 30- 50% reduction of the original reading, the sampler and skimmer cones should be cleaned.
Detector	A detector scan is performed quarterly and voltage optimized if necessary. Place scans in logbook. Maximum detector setting is approximately 2700-3000 volts.

Air Filter Air filter is located behind panel 1. Wash filter with tap water or

vacuum with shop vac as needed.

SHUTDOWN

Note: ICP-MS mains instrument power, PC power, exhaust fans, recirculator and vacuum system

remain on continuously.

Flush the system by aspirating 1 or 2% nitric acid for a few minutes, then aspirating di

water for a few minutes.

Turn plasma off.

Disengage pressure bars from peristaltic pump tubing.

TABLE 9. SPIKING CONCENTRATIONS

Element	Stock concentrations (ug/mL)	Spike amt. Water matrix (ug/L)	Spike amt. soil matrix (mg/kg)
Al	2000	4000	*
Sb	500	1000	100
As	2000	4000	400
Ba	2000	4000	400
Be	50	100	10
Cd	50	100	10
Ca	*	*	*
Cr	200	400	40
Co	500	1000	100
Cu	250	500	50
Fe	1000	2000	*
Pb	500	1000	100
Mg	*	*	*
Mn	500	1000	100
Mo	1000**	500	50
Ni	500	1000	100
K	*	*	*
Se	2000	4000	400
Ag	50	100	10
Na	*	*	*
Tl	2000	4000	400
Th	1000**	500	100
Sn	1000**	500	100
Ti	1000**	500	100
U	1000**	500	100
V	500	1000	100
Zn	500	1000	100

these concentrations are obtained by diluting 100 uL of each of Inorganic Ventures, Inc. Cat# CLPP-SPK-SET for water matrix with 50mL final volume. 100 uL of each of Inorganic Ventures, Inc. Cat# CLPP-SPK-SET for soil matrix with 50mL final volume.

* these analytes are not routinely spiked due to the typically large indigenous concentrations in
samples.
** these analytes are not present in the above mentioned three-spike-set and are therefore spiked
at specified levels using single element standards.

NOTE: For direct analyses (turbidity NTU<1, method 200.8) analytes are commonly spiked at 200
ug/L using standards from table 4.

NOTE: Spike levels may be modified at analyst discretion.

TABLE 10. ACCEPTANCE LIMITS FOR MATRIX SPIKES, SPIKE DUPLICATES AND LCS*

Element	Applicable Water Methods+	Water Accuracy %R	Water Precision RPD	Applicable Soil Methods+	Soil Accuracy %R	Soil Precision RPD
Al	+	75-125	0-20	+	75-125	0-20
Sb	+	75-125	0-20	+	75-125	0-20
As	+	75-125	0-20	+	75-125	0-20
Ba	+	75-125	0-20	+	75-125	0-20
Be	+	75-125	0-20	+	75-125	0-20
Cd	+	75-125	0-20	+	75-125	0-20
Ca	+	75-125	0-20	+	75-125	0-20
Cr	+	75-125	0-20	+	75-125	0-20
Co	+	75-125	0-20	+	75-125	0-20
Cu	+	75-125	0-20	+	75-125	0-20
Fe	+	75-125	0-20	+	75-125	0-20
Pb	+	75-125	0-20	+	75-125	0-20
Mg	+	75-125	0-20	+	75-125	0-20
Mn	+	75-125	0-20	+	75-125	0-20
Mo	+	75-125	0-20	+	75-125	0-20
Hg	+	75-125	0-20	+	75-125	0-20
Ni	+	75-125	0-20	+	75-125	0-20
K	+	75-125	0-20	+	75-125	0-20
Se	+	75-125	0-20	+	75-125	0-20
Ag	+	75-125	0-20	+	75-125	0-20
Na	+	75-125	0-20	+	75-125	0-20
Tl	+	75-125	0-20	+	75-125	0-20
Th	+	75-125	0-20	+	75-125	0-20
Sn	+	75-125	0-20	+	75-125	0-20
Ti	+	75-125	0-20	+	75-125	0-20
U	+	75-125	0-20	+	75-125	0-20
V	+	75-125	0-20	+	75-125	0-20
Zn	+	75-125	0-20	+	75-125	0-20

\+ See table 1 for analyte approval/ method acceptability status
* LCS limits for Method 6020 are 75-125%

TABLE 11. TYPICAL RUN SEQUENCE

When considering all of the aforementioned quality assurance requirements, the following run sequence becomes apparent from an operational point of view.

Instrument warm-up (30-60 minutes)
Perform mass calibration
Conduct a performance test
Calibration blank
Calibration standard 1
Calibration standard 2
Calibration standard 3
Initial Calibration Verification
Initial Calibration Blank
Interference Check Solution A (method 6020 only)
Interference Check Solution AB (method 6020 only)
sample or qc sample n
sample or qc sample n+1
sample or qc sample n+2
sample or qc sample n+3
sample or qc sample n+4
sample or qc sample n+5
sample or qc sample n+6
sample or qc sample n+7
CCV1
CCB1
sample or qc sample n+8
sample or qc sample n+9
sample or qc sample n+10
sample or qc sample n+11
sample or qc sample n+12
sample or qc sample n+13
sample or qc sample n+14
sample or qc sample n+15
sample or qc sample n+16
sample or qc sample n+17
CCV2
CCB2
sample or qc sample n+18
.
.
.
CCVfinal
CCBfinal

TABLE 12. ICP-MS MULTI-LABORATORY PRECISION AND ACCURACY DATA FOR AQUEOUS SOLUTIONS

Element	Comparability Range[a]	%RSD Range	N[b]	S[c]
Al	95-100	11-14	14-14	4
Sb	[d]	5.0-7.6	16-16	3
As	97-114	7.1-48	12-14	4
Ba	91-99	4.3-9.0	16-16	5
Be	103-107	8.6-14	13-14	3
Cd	98-102	4.6-7.2	18-20	3
Ca	99-107	5.7-23	17-18	5
Cr	95-105	13-27	16-18	4
Co	101-104	8.2-8.5	18-18	3
Cu	85-101	6.1-27	17-18	5
Fe	91-900	11-150	10-12	5
Pb	71-137	11-23	17-18	6
Mg	98-102	10-15	16-16	5
Mn	95-101	8.8-15	18-18	4
Ni	98-101	6.1-6.7	18-18	2
K	101-114	9.9-19	11-12	5
Se	102-107	15-25	12-12	3
Ag	104-105	5.2-7.7	13-16	2
Na	82-104	24-43	9-10	5
Tl	88-97	9.7-12	18-18	3
V	107-142	23-68	8-13	3
Zn	93-102	6.8-17	16-18	5

[a] Comparability refers to the percent agreement of mean ICP-MS values to those of the reference technique.
[b] N is the range of the number of ICP-MS measurements where the analyte values exceed the limit of quantitation (3.3 times the average IDL value).
[c] S is the number of samples with results greater than the limit of quantitation.
[d] No comparability values are provided for antimony because of evidence that the reference data is affected by an interference.

TABLE 13. ICP-MS MULTI-LABORATORY PRECISION AND ACCURACY DATA FOR SOLID MATRICES

Element	Comparability Range[a]	%RSD Range	N[b]	S[c]
Al	83-101	11-39	13-14	7
Sb	[d]	12-21	15-16	2
As	79-102	12-23	16-16	7
Ba	100-102	4.3-17	15-16	7
Be	50-87	19-34	12-14	5
Cd	93-100	6.2-25	19-20	5
Ca	95-109	4.1-27	15-17	7
Cr	77-98	11-32	17-18	7
Co	43-102	15-30	17-18	6
Cu	90-109	9.0-25	18-18	7
Fe	87-99	6.7-21	12-12	7
Pb	90-104	5.9-28	15-18	7
Mg	89-111	7.6-37	15-16	7
Mn	80-108	11-40	16-18	7

Ni	87-117		9.2-29		16-18		7	
K	97-137		11-62		10-12		5	
Se	81	39		12		1		
Ag	43-112		12-33		15-15		3	
Na	100-146		14-77		8-10		5	
Tl	91	33		18		1		
V	83-147		20-70		6-14		7	
Zn	84-124		14-42		18-18		7	

[a] Comparability refers to the percent agreement of mean ICP-MS values to those of the reference technique.

[b] N is the range of the number of ICP-MS measurements where the analyte values exceed the limit of quantitation (3.3 times the average IDL value).

[c] S is the number of samples with results greater than the limit of quantitation.

[d] No comparability values are provided for antimony because of evidence that the reference data is affected by an interference.

TABLE 14. CALCULATIONS

Parameter	Equation	Units
Water concentration	$(C_x)\ (d)$ where : C_x = concentration of the element being measured from the calibration curve in µg/L d = dilution factor	µg/L
Soil concentration (dry weight)	$\frac{(C_x)\ (V_t)\ (d)}{(W_s)}$ where : C_x = concentration of the element being measured from the calibration curve in µg/L W_s = initial dry weight of sample in Kg d = dilution factor V_t = final volume of digestate in liters	mg/Kg
Recovery (%) for ICV, CCV, LCS	$\frac{(C_x)}{(S)} * 100$ where : C_x = concentration of the element being measured from the calibration curve in µg/L or mg/Kg S = spike value in µg/L or mg/Kg	µg/L or mg/Kg
Recovery (%) for MS, MSD	$\frac{(C_x) - (S)}{(S)} * 100$ where : C_x = concentration of the element being measured from the calibration curve in µg/L or mg/Kg S = spike value in µg/L or mg/Kg	µg/L or mg/Kg
Relative Percent Difference (RPD)	$\frac{\lvert D1-D2\rvert}{(D1+D2)/2} * 100$ where : D1 = first sample value D2 = second sample value (replicate)	

TABLE 14. *(CONTINUED)* CALCULATIONS

Parameter	Equation	Units
Percent Solids	$\% \text{ solids} = \frac{\text{dry weight of sample (in kg)}}{\text{wet weight of sample (in kg)}} * 100$	
Percent Moisture	$\% \text{ moisture} = 100 - \% \text{ solids}$	

APPENDIX 1. AUTODUILUTER ACCESSORY

The autodiluter accessory located on the autosampler allows for both on-line dilution of over range samples and off-line dilution of samples prior to analysis.

On-line dilution actions are specified in the analytical method.

When using the diluter off-line it , run "Roboprep" application and follow instruction in software. This software is set up similar to the Ultramass software.

Verify that the diluter is being fed the correct diluent and there is adequate diluent for the samples in the sequence.

Start the sequence.

APPENDIX 2. MDL's and PQL's

ELEMENT		ISOTOPE	Water MDL	Water PQL	Soil MDL	Soil PQL
			ug/L	ug/L	mg/kg*	mg/kg
Al	Aluminum	27	6.1194	200	0.1583	10
Sb	Antimony	121	0.1682	100	0.0093	5
As	Arsenic	75	0.3315	50	0.2822	5
Ba	Barium	135soil	0.2176	5	0.2176	0.5
Ba	Barium	137water	0.3082	5	0.3082	0.5
Be	Beryllium	9	0.0605	4	0.1102	0.5
Cd	Cadmium	114	0.0723	5	0.1300	0.05
Cr	Chromium	52	0.1458	5	0.1669	0.5
Co	Cobalt	59	0.0686	5	0.0650	0.5
Cu	Copper	65	0.0686	25	0.2377	5
Pb	Lead	206,7,8	0.0966	1	0.0750	1
Mn	Manganese	55	0.0723	5	0.0994	0.5
Mo	Molybdenum	98	0.293	5	1.5007	5
Hg	Mercury	202	0.125	0.2	0.012	.1
Ni	Nickel	60	0.249	5	0.3004	0.5
Se	Selenium	78	0.4319	50	0.2782	5
Ag	Silver	107	0.0596	5	0.0953	0.5
Tl	Thallium	205	0.0730	5	0.0658	5
Th	Thorium	232	0.0521	5	0.3958	5
Sn	Tin	118	0.3277	5	0.777	5
Ti	Titanium	49	0.1751	5	0.296	5
U	Uranium	238	0.0614	5	0.1150	5
V	Vanadium	51	0.0435	5	0.0501	0.5
Zn	Zinc	66	0.5029	10	0.3693	2

* Soil MDL based on a sample size of 0.5g with a final volume of 50 mL.

GAM 6010A
Inductively Coupled Plasma-Atomic Emission Spectroscopy
Revision 3.11: 1/15/97

ANALYTES:		CAS #
Aluminum	(Al)	7440-36-0
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-43-9
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Lithium	(Li)	7439-93-2
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Phosphorous	(P)	7723-14-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Strontium	(Sr)	7440-24-6
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

INSTRUMENTATION: ICP-AES

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to the above listed analytes. The primary analyte wavelengths, method detection limits and reporting limits are given in table 1. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

1.2 Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Methods 3005A (GAM3005) or 3015 (GAM3015) for metals in water, Methods 3050A (GAM 3050A) or 3051 (GAM 3051) for metals in soil, 1311 (GAM1311) or 3015 (GAM3015) for TCLP, 1312 (GAM1312) or 3015 (GAM3015) for SPLP). When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 Method 6010A describes the sequential, multi-elemental determination of elements by ICP-AES. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photo multiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background photo-multiplier-correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 3.0 should also be recognized and appropriate corrections made; tests for their presence are described in step 8.3.

3.0 INTERFERENCES

3.1 Spectral interferences are caused by: (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuum or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternative wavelength. Background correction adjacent to the analyte line.

Users of all ICP instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Potential spectral interferences are given in table 2. The data in table 2 are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interfering elements can be assumed.

3.1.1 The interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696nm) in a sample containing approximately 10 mg/L of Al. According to table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in table 2.

3.1.2 The dashes in table 2 indicate that no measurable interferences were observed even at higher interferent concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

3.1.3 At present, information on the listed silver and potassium wavelengths is not available, but it has been reported that second-order energy from the magnesium 383.231-nm wavelength interferes with the listed potassium line at 766.490 nm.

3.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP-AES technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled argon plasma emission spectrometer: (Varian Liberty 100 I.D.# ICP100-2011144 coupled with a CETAC U-5000AT Ultrasonic Nebulizer I.D.#019206AT.)

4.1.1 Computer-controlled emission spectrometer with background correction. (Computer is a Premier Data Systems with a Grid 800 monitor. The background correction software offered by Varian, gives the analyst 5 different options. You can choose from a) POLYNOMIAL PLOTTED BACKGROUND where the software applies a background correction algorithm to estimate the background level at chosen wavelength position. (This is the background correction most frequently used), b) OFFPEAK BC- LEFT AND RIGHT where the user specifies the background points, c) OFFPEAK BC- LEFT ONLY where the user specifies a background point, d) OFFPEAK BC- RIGHT ONLY where the user specifies a background point, e) NONE where no background correction is required.

4.1.2 Radio frequency generator compliant with FCC regulations (40.86MHz).

4.1.3 Argon gas supply - Welding grade or better. (AGA 45 or 52 gallon liquid argon)

4.2 Operating conditions - Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where coordination factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results. Specific conditions have been established and are currently being used for the various analytical lines. Those conditions are chosen and saved within the Varian ICP software in the method library and are also given in table 3.

4.3 Class A volumetric flasks: 5-, 10-, 25-, 50-, and 100-mL.

4.4 Class A TD volumetric pipettes: 0.5-, 1.0-, 2.0-, 3.0-, 4.0-, 5.0-, and 10-mL.

4.5 MLA Precision air displacement pipetters: 10-, 25-, 50-, 100-, 250-, and 1000-uL.

4.6 Analytical balance- capable of accurate measurement to 0.01g.
(Sartorious PT 120 ID#30121328 (120g max))
(Mettler-Toledo PB602 (610g max))
Analytical balance- capable of accurate measurement to 0.0001g.
(Sartorious A200S)

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.(e.g.Mallinckrodt from VWR).
5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an SMI TopSider Series 2 liquid dispenser.
5.1.3 Nitric acid (conc), HNO3.(e.g.Tracepur Plus by EM from VWR).
5.1.4 Nitric acid (1:1), HNO3. Add 500 mL concentrated HNO3 to 400 mL water and dilute to 1 liter in an SMI Topsider Series 2 liquid dispenser.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water must meet ASTM type II standards. Reagent water will be interference free. Reagent water is dispensed from a Barnstead Nano-Pure unit Model #D4741, Serial #687920145027. Cartridges are changed approximately every six months. The Barnstead unit is fed water from a Kinetico commercial reverse osmosis unit (serial #361001). This unit has the capability to produce 75 gallons of water per day and has a 20 gallon bladder tank for storage. This unit also has a 10", 5 micron prefilter which is changed every other time the cartridges are changed.

5.3 Standard stock solutions may be purchased from a number of different manufacturers. The manufacturers commonly used are as follows: a)Inorganic Ventures b)Environmental Express c)Ultra Scientific. All catalogues from manufacturers are accessible in the inorganic laboratory. Table 4 gives a list of common concentrations sold by most manufacturers, common dilution concentrations used in this laboratory for calibration purposes and common calibration verification concentrations.

5.4 Mixed calibration standard solutions - Mixed calibration standard solutions may be prepared by combining appropriate volumes of the stock solutions using volumetric pipettes. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging. Calibration standards must be initially verified using a quality control sample and monitored weekly for stability. Some typical calibration

standard combinations are listed in table 5. All mixtures should then be scanned using a sequential spectrometer to verify the absence of inter-element spectral interference in the recommended mixed standard solutions.

[NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. Depending on the combination and concentration of acids used, the silver concentration should be minimal. Higher concentrations of silver require the addition of, or additional HCl.]

5.5 Two types of blanks are required for the analysis. The calibration blank is used in establishing the analytical curve, and the reagent blank is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

5.5.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. Other names used for this blank are the initial calibration blank and the continuing calibration blank. This sample is analyzed prior to an analytical sequence run, after every 10 samples, and at the end of an analytical run (see section 8.4 for use).

5.5.2 The reagent blank must contain all the reagents and in the same volumes as used in the processing of the samples. Other name used for this blank are the prep blank and the method blank. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis (see section 8.1 for use).

5.6 The instrument check standard is prepared by the analyst by combining compatible elements at concentrations which fall within their respective calibration curves. Other names for this standard are the initial calibration verification and the continuing calibration verification. This standard is prepared by the analyst from stock standards from a source different than that of the calibration standard (see section 8.4 for use).

5.7 The interference check solutions are prepared by diluting multi-element interferent check standards purchased from Environmental Express. The first standard contains known concentrations of interfering elements that will provide an adequate test of the correction factors. The second standard contains elements of interest in concentrations at least 10 times the instrumental detection limits. Table 6 contains a list of the elements within the interferent check standards and their concentrations along with the diluted concentrations. Within an analytical run standard #1 must be diluted and analyzed by itself (contains interfering elements) and immediately following standards #1+#2 must be diluted, mixed together and analyzed (see step 8.4.2).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous samples shall be preserved to pH <2 with nitric acid and have a holding time of 6 months for analysis of all metals except mercury which has a holding time of 28 days. Soil samples have a holding time of 6 months for all metals except mercury which has a holding time of 28 days.

7.0 PROCEDURE

7.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Solubilization and digestion procedures are presented in sample preparation methods 3005A(GAM3005A)/3015(GAM3015), 3050A(GAM3050A)/3051(GAM3051A), 1311(GAM1311), 1312(GAM1312).

7.2 Review/follow the daily setup and maintenance procedures in table 7.

7.3 Set up the instrument with proper operating parameters established in Step 4.2. The instrument must be allowed to become thermally stable before beginning (requiring 60 minutes of warm-up prior to calibration).

7.4 Calibrate the instrument using a calibration standard solution (see Step 5.3, 5.4), and a calibration blank (5.5.1). (Use the average intensity of three exposures for both standardization and sample analysis to reduce random error.)

7.5 Before beginning the sample run, re-analyze the highest mixed calibration standard as if it were a sample. Concentration values obtained may not deviate from the actual values by more than 5%. If the values differ by more than 5%, re-analyze the standard again. If, again, the values differ by more than 5% the instrument must be recalibrated.

7.6 Flush the system with the calibration blank solution for at least 30 seconds (step 5.5.1) before the analysis of each sample, standard, blank, or check solution.

7.7 Before beginning the sample run, re-analyze the calibration blank as a sample. The calibration blank must meet QC requirements and run frequencies as specified in section 8.4.1.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent. See table 8 for spiking concentrations.

8.1.1 Calculate the values for the preparation blanks, laboratory control samples (LCS), matrix spikes(MS) and matrix spike duplicates(MSD)

8.1.1.1 If the preparation blank shows contamination at a level of less than the reporting limit, or less than 10% of the concentration of the analyte present in sample, the method is presumed in control and sample analysis can proceed.

8.1.1.2 If the preparation blank contains contamination above the reporting limit, and greater than 10% of the concentration of the analyte of interest present in the samples associated with the blank, corrective actions must be performed to bring the method back into control. After the corrective actions are performed the analyst(s) must demonstrate that the preparation and analysis procedures are free of contaminants before sample analysis can proceed.

8.1.1.3 Calculate the spike recoveries for the LCS, MS and MSD. If all recoveries are within the established

limits in table 9 the method is presumed in control and sample analysis can proceed.

8.1.1.4 If the spike recoveries for the LCS are within the established control limits in table 9, but the MS (and/or MSD) are not within the established limits in table 9, the method is presumed in control and sample analysis can proceed. Sample data for the spiked sample with recoveries outside of the acceptance limits in table 9 should be flagged as "estimated concentration."

8.1.1.5 If the spike recoveries for the LCS are not within the established control limits in table 9, corrective actions must be performed to bring the method back into control. After corrective actions are performed, the analyst(s) must demonstrate LCS recoveries within the established limits before sample analysis can proceed.

8.2 Dilute and reanalyze samples that are more concentrated than the linear dynamic range.

8.3 Two additional tests are performed as required prior to reporting concentration data for analyte elements. These tests, as outlined in Steps 8.3.1 and 8.3.2, will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

8.3.1 Serial dilution: Perform a serial dilution to the same sample that a pre digestion matrix spike and spike duplicate were performed on if the pre digestion spike and spike duplicate fell outside of acceptable limits (see table 9). If the pre digestion matrix spike or matrix spike duplicate was recovered within acceptable limits this step is eliminated. If the LCS failed, this step is not necessary as the samples must be redigested. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the reporting limit after dilution), an analysis of a 1:5-dilution should agree within +/- 10% of the original determination. If not, the data should be flagged as estimated concentration.

8.3.2 Post Digestion Matrix spike addition: Perform a post digestion matrix spike addition to the same sample that a pre digestion matrix spike and spike duplicate were performed on if the pre digestion spike and spike duplicate fell outside of acceptable limits (see table 9). If the pre digestion matrix spike or matrix spike duplicate was recovered within acceptable limits this step is eliminated. If the LCS failed, this step is not necessary as the samples must be redigested. An analyte spike added to a portion of a prepared sample, or its dilution should be recovered to within 75% to 125% of the known value. The spike addition concentration is determined by the analyst based on the amount of analyte already present in the sample. If the spike is not recovered within the specified 75% to 125% limits, the data is flagged as estimated concentration.

8.4 Check the instrument standardization by analyzing appropriate check standards as follows.

8.4.1 Verify calibration every 10 samples and at the end of the analytical run, using a calibration blank (a.k.a.:ICB,CCB Step 5.5.1) and a check standard (a.k.a.:ICV,CCV Step 5.6).

8.4.1.1. The results of the check standard are to agree within 10% of the expected value; if not, rerun the instrument check standard. If it is not within 10% for a second time terminate the analysis, correct the problem, and reanalyze the samples associated with that check standard.

8.4.1.2 The results of the calibration blank are to agree within three standard deviations of the mean blank value. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, and reanalyze the samples associated with that calibration blank.

8.4.2 Analyze the interference check sample (Step 5.7) at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent. Results should be within +/- 20% of the true value.

8.5 Corrective Actions

8.5.2 If the laboratory control sample (LCS) is out of control the following procedures are required.

8.5.2.1 Check to be sure there are no errors in calculations, spiking solutions. Also, check instrument performance. You may use the criteria in section 8.2 above.

8.5.2.2 Recalculate the data and/or re analyze the sample if any of the above checks reveal a problem.

8.5.2.3 Re-analyze or re-digest and re-analyze the LCS sample to demonstrate that the analysis is in control.

8.5.2.4 Re-digest and re-analyze all samples associated with the unacceptable LCS.

8.5.3 Samples that are prepared and run with an out of control preparation blank must be re-digested and re-run along with a new preparation blank.

8.5.4 Flag data from samples that have unacceptable pre- or post-matrix spike recoveries as estimated concentration.

8.5.5 Flag data from samples that have unacceptable serial dilution recoveries as estimated concentration.

9.0 METHOD PERFORMANCE

9.1 In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table 10 lists the true values, the mean reported values, and the mean percent relative standard deviations.

9.2 In a single laboratory evaluation, seven wastes were analyzed for 22 elements by this method. The mean percent relative standard deviation from triplicate analyses for all elements and wastes was 9 +/- 2%. The mean percent recovery of spiked elements for all wastes was 93 +/- 6%. Spike levels ranged from 100 ug/L to 100 mg/L. The wastes included sludges and industrial wastewaters.

10.0 REFERENCES

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6. Rohrbough, W.G.; et al. Reagent Chemicals. American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.
7. 1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

TABLE 1. PRIMARY WAVELENGTHS, METHOD DETECTION LIMITS, AND REPORTING LIMITS

Elements	Wavelength(a)(nm)			MDLsoil* (ppm)	MDLwater (ppm)	RLsoil (ppm)	RLwater (ppm)
Aluminum	396.152	II	S	1.6226	0.0144	10.0	0.200
Antimony	206.833	I	H	3.8362	0.0043	5.00	0.100
Arsenic	188.979	I	H	4.7081	0.0324	5.00	0.050
Arsenic	193.696	I	H	2.9031	0.0176	5.00	0.050
Barium	455.403	II	S	0.1055	0.0006	0.50	0.005
Beryllium	313.042	II	S	0.1101	0.0011	0.50	0.005
Cadmium	228.802	I	H	0.1804	0.0017	0.50	0.005
Calcium	317.933	II	S	6.9356	0.0277	250.	5.00
Chromium	267.716	II	H	0.0949	0.0015	0.50	0.005
Cobalt	228.616	II	H	0.1569	0.0008	0.50	0.005
Copper	324.754	I	S	0.7502	0.0012	1.00	0.005
Iron	259.940	II	H	2.3898	0.0036	5.00	0.100
Lead	220.353	II	H	0.6350	0.0039	1.00	0.005
Lithium	670.784	I	S	0.1051	0.0002	0.50	0.005
Magnesium	279.553	II	H	5.5873	0.0185	250.	5.00
Manganese	257.610	II	H	0.3116	0.0007	0.50	0.005
Molybdenum	202.030	I	H	0.4657	0.0021	1.00	0.005
Nickel	231.604	II	H	0.3670	0.0035	0.50	0.005
Potassium	766.491	I	S	4.6544	0.0270	250.	5.00
Selenium	196.026	I	H	3.9136	0.0184	5.00	0.050
Silver	328.068	I	S	0.1589	0.0009	0.50	0.005
Sodium	588.995	I	S	8.8259	0.0438	250.	5.00
Thallium	190.864	I	H	2.3151	0.0193	5.00	0.050
Vanadium	292.402	II	H	0.4384	0.0013	0.50	0.005
Zinc	213.856	I	H	1.7503	0.0009	2.00	0.010

I = represent Atomic Wavelengths H = represent 'hard' Wavelengths
II = represent Ionic Wavelengths S = represent 'soft' Wavelengths

Atomic Wavelengths are those originating from the atomic state "I" whereas those originating from the ionic state are defined as ionic "II". Further, because there is a large spread in the transitional energies for different elemental wavelengths, the wavelengths can be characterized into groups defined as hard (e.g. those below 300nm) and soft wavelengths (e.g. those above 300nm).

* MDLsoil is based on a 0.5 gram sample size.

TABLE 2. ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM INTERFERENCE AT THE 100-mg/L LEVEL

		Interferent(a,b)									
Analyte	(nm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenu	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

(a) Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L
Ca - 1000 mg/L
Cr - 200 mg/L
Cu - 200 mg/L
Fe - 1000 mg/L
Mg - 1000 mg/L
Mn - 200 mg/L
Tl - 200 mg/L
V - 200 mg/L

(b) The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

BLE 3. OPERATION CONDITION PARAMATERS

Conditions	Settings
Viewing Height	6-15mm
Search Window	0.020-0.080nm
Integration Time	2-3.5 seconds
Replicates	3
PMT Voltage	650-800 V
RF Power	1.00-1.5 L/min.
Plasma argon flow	15.0 L/min.
Auxiliary flow	1.50 L/min.
Peristaltic pump speed	15.0 rpm
Nebulizer pressure	170 kPa
Stabilization time	10 sec.
Rinse time	10 sec.
Sample delay	20 sec.
Background correction	Polynomial Plotted

Variations in settings are due to different analytes being optimized at varying conditions. Settings are also matrix dependent.

TABLE 4. COMMON STOCK STANDARD CONCENTRATIONS AND CALIBRATION CONCENTRATIONS.

Element	**Common stock concentrations	**Common calibration concentrations	***Common calibration verification concentrations
Al	1,000 ug/ml	1.0, 10.0 ug/ml	1.0, 10.0 ug/ml
Ag	1,000 ug/ml	0.5 ug/ml	0.5 ug/ml
As	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Ba	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Be	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Ca	1,000 ug/ml	1.0, 10.0 ug/ml	1.0, 10.0 ug/ml
Cd	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Co	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Cr	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Cu	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Fe	1,000 ug/ml	1.0, 10.0 ug/ml	1.0, 10.0 ug/ml
K	1,000 ug/ml	1.0, 10.0 ug/ml	1.0, 10.0 ug/ml
Mg	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Mn	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Mo	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Na	1,000 ug/ml	1.0, 10.0 ug/ml	1.0, 10.0 ug/ml
Ni	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Pb	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Sb	1,000 ug/ml	1.0, 10.0 ug/ml	0.5, 1.0 ug/ml
Se	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Tl	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
V	1,000 ug/ml	1.0 ug/ml	1.0 ug/ml
Zn	1,000 ug/ml	1.0, 10.0 ug/ml	1.0, 10.0 ug/ml

*these concentrations are common for most manufacturers to produce, but are not always the specific concentration produced.

**these concentrations represent common diluted concentrations used in this laboratory for instrument calibration purposes, but these may vary at any time depending on the matrix of the sample being analyzed and the concentration of the element of concern.

***these concentrations represent common diluted concentrations used in this laboratory for calibration verification purposes, but these may also vary at any time dependent of sample matrix and concentration of element of concern.

TABLE 5. RECOMMENDED MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As, Mo
IV	Al, Ca, Cr, K, Na, Ni, Li,& Sr
V	Ag (see Note to Step 5.4), Mg, Sb, and Tl
VI	P

TABLE 6. INTERFERENCE CHECK SOLUTION.

Element	INT-1(cat.#ICINT100-4) stock concentrations	INT-2(cat.#ICINT100-18) stock concentrations	*INT-1 diluted concentrations	**INT-1 + INT-2 diluted concentrations
Al	1200 ug/ml		12 ug/ml	12 ug/ml
Ca	6000 ug/ml		60 ug/ml	60 ug/ml
Fe	5000 ug/ml		50 ug/ml	50 ug/ml
Mg	3000 ug/ml		30 ug/ml	30 ug/ml
Na	1000 ug/ml		10 ug/ml	10 ug/ml
As		1,000 ug/ml		1 ug/ml
Ba		300 ug/ml		0.3 ug/ml
Be		100 ug/ml		0.1 ug/ml
Cd		300 ug/ml		0.3 ug/ml
Cr		300 ug/ml		0.3 ug/ml
Co		300 ug/ml		0.3 ug/ml
Cu		300 ug/ml		0.3 ug/ml
Pb		1,000 ug/ml		1.0 ug/ml
Mn		200 ug/ml		0.2 ug/ml
Hg		50 ug/ml		0.05 ug/ml
Ni		300 ug/ml		0.3 ug/ml
K		20,000 ug/ml		20 ug/ml
Se		500 ug/ml		0.5 ug/ml
Ag		300 ug/ml		0.3 ug/ml
Tl		1,000 ug/ml		1.0 ug/ml
V		300 ug/ml		0.3 ug/ml
Zn		300 ug/ml		0.3 ug/ml

*these concentrations are obtained by diluting Int-1 1:100 (1ml Int-1 into 100ml volumetric flask, bring to volume with appropriate acid concentrations and deionized water)

**these concentrations are obtained by diluting Int-1 1:50 with Int-2 1:1000 (2ml INT-1 + 100ul INT-2 into 100 ml volumetric flask, bring to volume with appropriate acid concentrations and deionized water)

TABLE 7. DAILY SETUP/SHUTDOWN AND MAINTENANCE PROCEDURES

DAILY SETUP

Note: ICP instrument power and PC power remain on continuously.
Turn on RF power.
Turn on recirculator.
Turn on USN chiller unit.
Perform any maintenance necessary (see MAINTENANCE section below).
If torch was replaced, set torch at proper height using torch tool.
Turn on exhaust fan.
Attach peristaltic pump tubing and USN drain pump tubing.
Light plasma.
Allow instrument to become thermally stable by allowing it to warm up for 30-60 minutes.
Perform a wavelength calibration.
Perform a horizontal torch alignment if torch was replaced.

MAINTENANCE

Recirculator	Check water level in recirculator (bimonthly). If indicator light is red or the instrument software repeatedly displays a high water conductivity error message, change the filter (annually or as needed).
	Check internal motor oil level annually.
	Vacuum ventilation fins (as needed).
USN chiller unit	Check coolant level- top off if necessary (level should be approximately one inch from top of reservoir).
	Drain and replace coolant (annually).
	Vacuum ventilation fins (as needed).
USN	Replace tubing that is worn or has deposits, dirt, debris that may restrict solution flow and affect instrument performance (daily or as needed).
	Visually inspect glass chamber during operation to assure a robust cloud of aerosol is being produced by the transducer (daily).
	Check that tubing from USN to torch contains no precipitation (daily before use).
Spray Chamber	Inspect for deposits, dirt, debris that may restrict solution flow and affect instrument performance (daily).
	Replace o-rings (as necessary).
Concentric Nebulizer	Inspect for deposits, dirt, debris that may restrict solution flow and affect instrument performance (daily).
Gas Supply	Check Ar level (daily). If level becomes too low, the instrument software will display a low argon pressure error message.
Torch/bonnet	Inspect for deposits, dirt, debris that may impair emissions measurements and affect instrument performance (daily).
ICP	Clean window in torch compartment (monthly or as needed).
	Vacuum ventilation fins (as needed).
	Keep external cabinet clean (daily).
	Inspect coil for signs of deterioration/corrosion.

SHUTDOWN

Note: ICP instrument power and PC power remain on continuously.
Flush the system by aspiration 5% nitric acid for a few minutes, then aspirating DI water for a few minutes.
Turn plasma off.
Turn off RF supply.
Turn off USN (operate button, then power button).
Turn off USN chiller unit.
Turn off recirculator.
Turn off exhaust fan.
Release peristaltic pump tubing and drain pump tubing on USN.

TABLE 8. SPIKING CONCENTRATIONS

Element	Stock concentrations (ug/mL)	Spike amt. water matrix (ug/L)	Spike amt. soil matrix (mg/kg)
Al	2000	4000	*
Sb	500	1000	200
As	2000	4000	800
Ba	2000	4000	800
Be	50	100	20
Cd	50	100	20
Ca	*	*	*
Cr	200	400	80
Co	500	1000	200
Cu	250	500	100
Fe	1000	2000	*
Pb	500	1000	200
Mg	*	*	*
Mn	500	1000	200
Mo			
Hg			
Ni	500	1000	200
K	*	*	*
Se	2000	4000	800
Ag	50	100	20
Na	*	*	*
Tl	2000	4000	800
V	500	1000	200
Zn	500	1000	200

these concentrations are obtained by diluting 100 uL for water matrix with 50mL final volume
200 uL for water matrix with 100mL final volume
200 uL for soil matrix with 50mL final volume
400 uL for soil matrix with 100mL final volume
each of the three-spike-set from Inorganic Ventures, Inc. Cat# CLPP-SPK-SET.

* these analytes are not spiked due to the typically large indigenous concentrations in samples.

TABLE 9. ACCEPTANCE LIMITS FOR MATRIX SPIKES, SPIKE DUPLICATES AND LCS*

Element	Water methods (EPA SW846)	Water Accuracy %R	Water Precision RPD	Soil methods (EPA SW846)	Soil Accuracy %R	Soil Precision RPD
Al	6010A	75-125	0-20	6010A	75-125	0-20
Sb	6010A	75-125	0-20	6010A	75-125	0-20
As	6010A	69-116	0-20	6010A	56-127	0-20
Ba	6010A	67-118	0-20	6010A	51-125	0-20
Be	6010A	75-125	0-20	6010A	75-125	0-20
Cd	6010A	67-115	0-20	6010A	60-119	0-20
Ca	6010A	75-125	0-20	6010A	75-125	0-20
Cr	6010A	67-110	0-20	6010A	65-104	0-20
Co	6010A	75-125	0-20	6010A	75-125	0-20
Cu	6010A	75-125	0-20	6010A	75-125	0-20
Fe	6010A	75-125	0-20	6010A	75-125	0-20
Pb	6010A	68-111	0-20	6010A	60-108	0-20
Mg	6010A	75-125	0-20	6010A	75-125	0-20
Mn	6010A	75-125	0-20	6010A	75-125	0-20
Mo	6010A	75-125	0-20	6010A	75-125	0-20
Hg	7470	71-109	0-20	7471	60-117	0-20
Ni	6010A	75-125	0-20	6010A	75-125	0-20
K	6010A	75-125	0-20	6010A	75-125	0-20
Se	6010A	70-123	0-20	6010A	61-137	0-20
Ag	6010A	61-123	0-20	6010A	70-120	0-20
Na	6010A	75-125	0-20	6010A	75-125	0-20
Tl	6010A	75-125	0-20	6010A	75-125	0-20
V	6010A	75-125	0-20	6010A	75-125	0-20
Zn	6010A	75-125	0-20	6010A	75-125	0-20

*All LCS samples have interim acceptance limits of 75-125%

TABLE 10. ICP PRECISION AND ACCURACY DATA(a)

Element	Sample No. 1 True Value (ug/L)	Sample No. 1 Mean Reported Value (ug/L)	Sample No. 1 Mean SD(b) (%)	Sample No. 2 True Value (ug/L)	Sample No. 2 Mean Reported Value (ug/L)	Sample No. 2 Mean SD(b) (%)	Sample No. 3 True Value (ug/L)	Sample No. 3 Mean Reported Value (ug/L)	Sample No. 3 Mean SD(b) (%)
Be	750	733	6.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	749	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	60	63	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	594	3.0	20	19	15	180	178	6.0
Al	700	696	5.6	60	62	33	160	161	13
Cd	50	48	12	2.5	2.9	16	14	13	16
Co	700	512	10	20	20	4.1	120	108	21
Ni	250	245	5.8	30	28	11	60	55	14
Pb	250	236	16	24	30	32	80	80	14
Zn	200	201	5.6	16	19	45	80	82	9.4
Se(c)	40	32	21.9	6	8.5	42	10	8.5	8.3

(a) Not all elements were analyzed by all laboratories.
(b) SD = standard deviation.
(c) Results for Se are from two laboratories.

Parameter	Equation	Units
Water concentration	$(C_x)\ (d)$ where : C_x = concentration of the element being measured from the calibration curve in mg/L d = dilution factor	mg/L
Soil concentration (dry weight)	$\frac{(C_x)\ (V_t)\ (d)}{(W_s)}$ where : C_x = concentration of the element being measured from the calibration curve in mg/L W_s = initial dry weight of sample in Kg d = dilution factor V_t = final volume of digestate in liters	mg/Kg
Relative Percent Difference (RPD)	$\frac{D1-D2}{(D1+D2)/2} * 100$ where : D1 = first sample value D2 = second sample value (replicate)	
Percent Solids	% solids = $\frac{\text{dry weight of sample}}{\text{wet weight of sample}}$ X100	percentage total

Attachment QAPP-B2

Soil Analysis of Mercury (Manual Cold Vapor Technique) Method 7471A

GAM: 7471A
Rev: 1.02
Date: 1-16-97
Pages: 8

Standard Operating procedure (SOP)

Method Name 7471A

Revised 9-27-99

Prepared by: _____ Date: 1-16-97
Analyst/Employee

Reviewed by: _____ Date: 1-16-97
Manager

Approved by: _____ Date: 1/16/97
QA Officer

GAM: 7471A
Rev: 1.0
Date: 9/27/99
Pages: 7

Standard Operating procedure (SOP)

Method Name 7471A

Prepared by: _____ Date: 9/27/99
Analyst/Employee

Reviewed by: _____ Date: _____
Manager

Approved by: _____ Date: _____
QA Officer

GAM 7471A
Soil Analysis Of Mercury (Manual Cold Vapor Technique)
Revision 1.0: 9/27/99

ANALYTE:	CAS#
Mercury (Hg)	7439-97-6

1. SCOPE AND APPLICATION

1.1 Determination of total mercury (inorganic + organic) in a soil matrix using the cold vapor atomic absorption technique.

1.2 The range of the method is 0.2 to 5.0 mg/Kg (ug/gram of Hg). The range may be changed by altering the sample aliquot size.

2. SUMMARY OF METHOD

2.1 Prior to analysis, soil samples are prepared (digested) to make soluble any inorganic mercury and convert organic forms of mercury to elemental mercury.

2.2 A cold vapor atomic absorption method is used based on elemental mercury absorbing radiation at a wavelength of 253.7 nm. Once the mercury is reduced to the elemental state, it is aerated into a closed system with a fixed cell path. The peak height of the absorbed radiation is measured as mercury concentration.

3. INTERFERENCES

3.1 Potassium permanganate is used to eliminate an interference from sulfide. Concentrations as high as 20 mg/L of sulfide (as sodium sulfide) are effectively removed by this technique.

3.2 Very high levels of copper (>10 mg/L) may interfere.

3.3 Samples with excessive chlorides may also interfere. Excess permanganate and additional hydroxylamine sulfate reagent may be used to counteract high chlorides.

3.4 Organic contaminants that absorb at 253.7 will be seen as an interference.

4. APPARATUS AND MATERIALS

4.1 Cetac M-6000A Automated Mercury Analyzer, with ASX-500 Random Access auto diluter. Software runs through a deskpro Compaq computer

4.2 300 mL BOD bottles, and plastic test tubes.

4.3 Volumetric pipettes 0.5, 1, 2, 4, 5, 10 Class "A" or equivalent.

4.4 MLA micro pipette from 10 ul to 1000 uL and disposable tips.

5. REAGENTS

5.1 5 % potassium permanganate = In a liter volumetric flask add 50 grams of granular reagent grade potassium permanganate to 500 mLs of deionized water. Bring to 1 liter final volume, add stirring bar and stir thoroughly on a stirring plate (approx. 15-20 minutes).

5.2 5 % potassium persulfate = In a liter volumetric flask add 50 grams of granular reagent grade potassium persulfate to 500 mls of deionized water. Bring to 1 liter final volume, add stirring bar and stir thoroughly on a stirring/heating plate. Add slight heat while stirring to allow total dissolvation of solids (approx. 20-30 minutes).

5.3 Sodium chloride-hydroxylamine sulfate = In a 1 liter volumetric flask, add 120 grams of granular reagent grade sodium chloride and 120 grams of granular reagent grade hydroxylamine sulfate to 500 mls of deionized water. Bring to 1 liter final volume add stirring bar and stir thoroughly on a stirring plateuntil totally dissolved.

5.4 Stannous chloride 10% = 100 grams of stannous chloride diluted in 1000 mLs with 70 mLs of HCl, and 930 mLs of D.I.

5.5 Aqua regia - prepare immediately before use by carefully adding three volumes of concentrated HCL to one volume of concentrated HNO3.

5.5.1 Hydrochloric acid - concentrated trace metals grade or equivalent.

5.5.2 Nitric acid - concentrated trace metals grade or equivalent.

5.6 Sulfuric acid - concentrated trace metals grade or equivalent.

5.7 Reagent water - ASTM Type II or better.

5.8 Mercury working standards

Working CAL standard A:	1 mL of 1,000 ug/mL Hg stock CAL standard diluted to 100 mL with d.i. water. Working CAL standard A should contain at least 1 mL of 1:1 nitric acid.
Working CAL standard B:	1 mL of 10 ug/mL Hg working CAL standard A diluted to 100 mL with d.i. water. Working CAL standard B should contain at least 1 mL of 1:1 nitric acid.
Working ICV standards:	Prepared as above using 1,000 ug/mL Hg stock ICV standard.

5.8.1. Standard stock solutions may be purchased from a number of different manufacturers. Commonly used manufacturers are as follows; a) Aldrich Chemical Co. b) Chem Service c) JT Baker (Baxter SP) d) Ricca Chemical Co. e) SPEX Industries F) Ultra Scientific.

6. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous samples shall be preserved to pH <2 with nitric acid and have a holding time of 6 months for analysis of all metals except mercury which has a holding time of 28 days. Soil samples have a holding time of 6 months for all metals except mercury which has a holding time of 28 days.

7.0 PROCEDURE

7.1 Instrument Calibration:

7.1.1 Prepare calibration standards on a daily basis by making serial dilutions as follows:

Working CAL standard A: 1 mL of 1,000 ug/mL Hg stock CAL standard diluted to 100 mL with d.i. water. Working CAL standard A should contain at least 1 mL of 1:1 nitric acid.

Working CAL standard B: 1 mL of 10 ug/mL Hg working CAL standard A diluted to 100 mL with d.i. water. Working CAL standard B should contain at least 1 mL of 1:1 nitric acid.

7.1.2 In 300 mL BOD bottles make the following calibration standards

7.1.3 A calibration curve must be prepared each day by transferring 0, 0.1, 0.5, 1.0, 2.0, 5.0, and 10.0 mL aliquots of working CAL Standard B to a series OF 100 mL. Adde 5 mLs of nitric to achieve a 5% HNO3 conc. to each volumetric, bring to volume with D.I. Proceed as indicated in 7.3 treating the standards in the same manner as samples.

Number of mL of Worikng CAL Standard B Added	Standard Concentration ug/mL	Amount in ug of Hg	Soil Concentration (based on 1g) mg/Kg
10	0.100	1.00	1.00
5	0.100	0.50	0.50
2	0.100	0.20	0.20
1	0.100	0.10	0.10
0.5	0.100	0.05	0.05
0.1	0.100	0.01	0.01
0	0.100	0.00	0.00

* Correlation coefficient must be 0.995 or better.

7.2 Calibration Verification

7.2.1 Prepare calibration verification standards on a daily basis by making serial dilutions as follows:

Working ICV standard A: 1 mL of 1,000 ug/mL Hg stock ICV standard diluted to 100 mL with d.i. water. Working ICV standard A should contain at least 1 mL of 1:1 nitric acid.

Working ICV standard B: 1 mL of 10 ug/mL Hg working ICV standard A diluted to 100 mL with d.i. water. Working ICV standard B should contain at least 1 mL of 1:1 nitric acid.

7.2.2 The ICV is run immediately after the calibration curve, followed by a calibration blank.

7.2.3 The ICV must be between 90-110% of expected value.

7.2.4 Calibration blank must be below the minimum reporting limit.

7.3 Weight out a representative 1 gram aliquot of the soil sample and place into a precleaned 300 mL BOD bottle and record the sample weight in the mercury cold vapor logbook. Label the BOD with the correct sample number using a permanent marker. All sample matrix spikes, duplicate spikes and blanks should be prepared by following the same procedures as the samples. Matrix spikes, duplicates and blanks are performed on each analytical batch or 20 samples whichever is more frequent. For matrix spiking concentrations and proper spiking techniques, see the procedure for cold vapor instrumentation setup and calibration.

7.4 Add 5 mLs of reagent water from the individually labeled polyethylene variable volume dispensers.

7.5 Add 5 mLs of aqua regia.

7.6 Place the BOD bottle into a water bath that has been preset at a temperature of 95C, for two minutes. The water bath must be preheated for at least 1 hour.

7.7 Carefully remove BOD from the water bath and place in the hood to be cooled.

7.8 After BOD bottle has cooled to room temperature, add 50 mLs of reagent water and 15 mLs potassium permanganate solution.

7.9 Place the BOD bottle into the water bath, preset at a temperature of 95C. Leave in the water bath for 30 minutes.

7.10 Carefully remove BOD from the water bath and place in the hood to be cooled.

7.11 After BOD bottle is cooled to room temperature, add 6 mls of sodium chloride-hydroxylamine sulfate solution, from the individually labeled polyethylene variable volume dispensers, to eliminate any excess potassium permanganate, and stir carefully by hand until purple color is gone.

7.12 Add 55 mL of reagent water to each BOD.

7.13 Using the air compressor, purge the head space (20 to 30 seconds) to remove possible gaseous interferences.

7.14 Filter digested samples prior to loading of samples onto instrument.

7.15 Continuing calibration standards, and ccb's need to be run after every 10 samples and at the end of each run. Criteria of plus or minus 20% on ccv'c and ccb's below detection the limit. This criteria must be met before sample analysis can proceed.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent.

8.2 Calculate the values for the preparation blanks, laboratory control samples (LCS), matrix spikes(MS) and matrix spike duplicates(MSD)

8.3 If the preparation blank shows no contamination above the reporting limits for the analytes of interest, the method is presumed in control and sample analysis can proceed.

8.4 If the preparation blank contains contamination above the reporting limit, corrective actions must be performed to bring the method back into control. After the corrective actions are performed the analyst(s) must demonstrate that the preparation and analysis procedures are free of contaminants before sample analysis can proceed.

8.5 Calculate the spike recoveries for the LCS, MS and MSD. If all recoveries are within the established limits in 9.5. The method is presumed in control and sample analysis can proceed.

8.6 If the spike recoveries for the LCS are within the established control limits in 9.5, but the MS (and/or MSD)are not within the established limits in 9.5, the method is presumed in control and sample analysis can proceed. Sample data for the spiked sample with recoveries outside of the acceptance limits in 9.5 should be flagged as "estimated concentration."

8.7 If the spike recoveries for the LCS are not within the established control limits in 9.5, corrective actions must be performed to bring the method back into control. After corrective actions are performed, the analyst(s) must demonstrate LCS recoveries within the established limits before sample analysis can proceed.

8.8 In 300 mL BOD bottles make the following calibration check and matrix spiking standards:

Standard Type	Number of mL of Working ICV Standard B Added	Standard Concentration ug/mL	Amount in ug of Hg	Soil Concentration (based on 1g) mg/Kg
Lab control sample (LCS)	4	0.100	0.40	0.400
Matrix spike	5	0.100	0.50	0.500
Matrix spike Duplicate	5	0.100	0.50	0.500
Calibration check	4	0.100	0.4	0.400
Continued Calibration check	4	0.100	0.40	0.400

8.9 Set up and maintenance, see table A.

Table A

The 1.0 The instrument is located next to the hood in the metals lab. Mercury vapor created will be vented into a KmnO4 trap so as not to be released into the atmosphere of the lab.

2.0 Turn the Argon gas on prior to instrument power up. Next, turn on the power for the analyzer, Hg lamp, and auto sampler, in that order. Open the software last, due to the fact that it will not boot properly if not done in this order.

3.0 Allow the instrument to to warm up for 2 hours prior to analysis setup.

3.1 Zero the instrument and read the highest standard by pressing the read icon on the analysis page. Press sample type "standard" then the highest standard number.

3.2 Zero the instrument. Set The 2 baseline corrections 4 seconds prior to the concave up of the Hg curve. Next, set the 2 time profile lines at the peak of the Hg curve. Each of these 2 parallel lines with 4 seconds between them.

4.0 Daily or after each use.
1. Immediately clean all spilled material.
2. Do not leave vapors in absorption cell after use.

9.0 Corrective Actions

9.1 Calculate recovery on all samples, blanks, spikes, duplicate spikes, and lab control samples. Determine if the recovery is within 75-112% of added concentration for MS and MSD. If recovery is not within limits, the following procedures are required.

9.2 Check to be sure there are no errors in calculations. Also, check instrument performance.

9.3 Recalculate the data and/or re-analyze the extract if any of the above checks reveal a problem.

9.4 Re-extract and re-analyze the sample. If none of the above are a problem, flag the data as "estimated concentration." All method and reagent blanks should be less than the MRL.

9.5 Laboratory control samples must have recoveries of 75-125%, matrix spikes, and matrix spike duplicates, must have recoveries of 60-117%.

9.6 RPD's should be 0-20% for matrix spikes and duplicates.

9.7 If the laboratory control sample (LCS) is out of control reprep the samples associated with the run.

9.8 Re-digest and re-analyze all samples associated with unacceptable blanks.

9.9 Re-digest and re-analyze all samples associated with unacceptable LCS recoveries.

9.10 Flag all sample data for unacceptable matrix spikes and matrix spike duplicate recoveries or RPD's as "estimated concentrations."

10.0 References

10.1 Mercury in solid or semi solid waste, EPA Method 7471A. September 1 1986

10.2 Mercury in solid or semi solid waste, SW 846 Method 7471A. September 1986

11.0

ELEMENT	Hg Mg/Kg
VALUE (SPIKE)	0.1000
S1	0.0856
S2	0.0842
S3	0.0777
S4	0.0784
S5	0.0772
S6	0.077
S7	0.075
MEAN	0.0793
STDEV	0.0040
MDL	0.0125

11.1 The MRL for mercury in soil is 0.1 ppb with RPD limits of 0-20%.

Attachment QAPP-B3

Analysis of Volatile Organic Hydrocarbons by GC/MS
Method 8260A

GAM 8260A

Analysis of Volatile Organic Hydrocarbons by Gas Chromatography/ Mass Spectrometry

Revision 6.4: 02/06/02

1.0 APPLICATION

Qualitative and quantitative determination of volatile organic compounds in wastewater and solid wastes, including soils, using purge and trap introduction to a gas chromatograph/mass spectrometer. The following method details the techniques used to identify and quantify 69 compounds using the SW-846 8260A methodology.

TARGET COMPOUNDS

COMPOUNDS	Retention Time	QUANT ION	SECONDARY ION # 1	SECONDARY ION # 2	METHOD DETECTION LIMIT
Dichlorodifluoromethane	4.10	85	87	101	5.0
Chloromethane	4.35	50+51	52	48	5.0
Vinyl chloride	4.46	62	64		2.0
Bromomethane	5.25	94	96		5.0
Chloroethane	5.29	49	63		5.0
Trichlorofluoromethane	5.51	101	103	105	5.0
1,1-Dichloroethene	6.40	61	96	63	5.0
Acetone	6.48	43	42		100.0
Carbon Disulfide	7.04	76	142		5.0
MTBE	7.35	73	57		5.0
Methylene chloride	7.44	49	47	51	5.0
trans-1,2-Dichloroethene	7.52	96	61	98	5.0
1,1-Dichloroethane	8.29	63	65	83	5.0
2,2-Dichloropropane	9.13	97	77		5.0
2-Butanone	9.16	43	39		100.0
Cis-1,2-Dichloroethene	9.35	96	61	98	5.0
1,1,1-Trichloroethane	10.00	97	99	130	5.0
Chloroform	10.05	83	85		5.0
1,1-Dichloropropene	10.11	75	77	110	5.0
Bromochloromethane	10.12	128+130			5.0
Carbon tetrachloride	10.16	117	119	121	5.0
Benzene	10.40	78	77		5.0
1,2-Dichloroethane d4 (surrogate)	11.00	65	67	102	5.0
1,2-Dichloroethane	11.08	62	98	64	5.0
Trichloroethene	11.43	130	132	95	5.0
1,2-Dichloropropane	12.13	63	61		5.0
Dibromomethane	13.00	93	95	174	5.0
Bromodichloromethane	13.03	83	85	127	5.0
cis-1,3-Dichloropropene	13.35	75	110	112	5.0
4-methyl-2-pentanone	13.21	85	43		100.0
Toluene d8 (surrogate)	13.46	98	100		5.0
Toluene	13.52	92	91		5.0
trans-1,3-Dichloropropene	14.43	75	110		5.0
Tetrachloroethene	14.51	166	168	129	5.0

2-Hexanone	14.57	43	58		100.0
1,3-Dichloropropane	15.15	76	78		5.0
1,1,2-Trichloroethane	15.21	83	85	87	5.0
Dibromochloromethane	16.15	129	127		5.0
1,2-Dibromoethane	16.16	107	109		2.0
Ethylbenzene	16.45	91	106		5.0
Chlorobenzene	16.53	112	114		5.0

*** note - Reporting limit for 5 mL or 5 gram (wet weight) sample.**

TARGET COMPOUNDS

[illegible] COMPOUNDS	[illegible]	QUANT. ION	SECONDARY ION (1)	SECONDARY ION (2)	METHOD REPORTING LIMIT (ug/L or ug/kg)
m-xylene	17.00	91	106		5.0
p-xylene	17.00	91	106		5.0
1,1,2,2-Tetrachloroethane	17.08	131	83	85	5.0
o-xylene	17.52	91	106		5.0
Styrene	18.05	104	78		5.0
Isopropylbenzene	18.31	105	120		5.0
Bromoform	19.13	173	175	171	5.0
n-Propylbenzene	19.18	91	120		5.0
Bromobenzene	19.34	156	77	158	5.0
1,3,5-Trimethylbenzene	19.41	105	120		5.0
2-Chlorotoluene	19.43	91	126		5.0
1,2,3-Trichloropropane	19.56	110	75		5.0
4-Chlorotoluene	19.57	91	127		5.0
2-Bromofluorobenzene (surrogate)	20.05	95	174	176	5.0
1,1,2,2-Tetrachloroethane	20.06	83	85		5.0
t-Butylbenzene	20.15	119	91		5.0
1,2,4-Trimethylbenzene	20.21	105	120		5.0
sec-Butylbenzene	20.36	105	134		5.0
4-Isopropyltoluene	20.52	119	134	91	5.0
1,3-Dichlorobenzene	21.12	146	148		5.0
1,4-Dichlorobenzene	21.31	146	148		5.0
n-Butylbenzene	21.34	91	134		5.0
1,2-Dichlorobenzene	22.08	146	148	111	5.0
1,2-Dibromo-3-chloropropane	23.48	75	155	157	5.0
Hexachlorobutadiene	24.49	225	260		5.0
1,2,4-Trichlorobenzene	24.48	180	182		5.0
Naphthalene	25.28	128			5.0
1,2,3-Trichlorobenzene	24.45	180	182		5.0

*** note-retention times will vary dependant upon instrument and column length.**
**** note - Reporting limit for 5 mL or 5 gram (wet weight) sample.**

2.0 DEFINITIONS

2.1 Purge And Trap - an extraction and concentration technique which uses inert gas to push volatile contaminants from a sample aliquot then traps them on a highly adsorbent material. This adsorbent trap is then flash heated to release the contaminants and introduce them directly into a gas chromatograph by changing the type of stationary phase, the flow rate of the mobile phase or by changing the temperature of the reactions taking place.

2.2 Mass Spectrometer - also known as a mass filter, the mass spectrometer functions by allowing only one mass per unit time to strike a detector known as an electron multiplier. Identification is performed by striking the compounds of interest with a beam of electrons in an ionization chamber. This electron beam has a fixed energy and results in the fragmentation or "ionization" of covalent bonds producing positive, negative and neutral charged ions. These ions are pushed into the mass filter using an electromagnetic force opposite in charge to the ions of interest, the most common type of ions used are the positive ions. Typically, the ions produced are focused using a series of electromagnetic lenses. The type and abundance of ions produced are characteristic chemical properties of the compound ionized. These ions are directly related to the covalent bond energies between the atoms in the compound. Compounds are identified using the characteristic "spectra" produced from the ionization process. All spectra are ratioed to the most abundant ion called the "base peak", the molecular weight of the compound can sometimes be determined by the presence of a molecule of the parent compound missing one electron (e-) which is called the parent ion. The technique is also enhanced by the presence of naturally occurring atomic isotopes which serve to identify not only the type of atom present in the compound but also the number of those atoms present in the compound.

2.3 Gas Chromatograph - an instrument which can separate complex mixtures of compounds using phase partitioning between a stationary phase and a mobile phase (i.e. - carrier gas). Each compound has an affinity for the stationary phase based on chemical properties specific to that compound such as boiling point, polarity, functional groups and molecular size. The greater the affinity for the stationary phase translates into a compounds increased residence time in that phase. The ratio of time spent in the stationary phase versus the time the compound spends in the mobile (or gas) phase gives us the elution time or better known as the retention time. Retention times can be changed for the same compound by changing the type or polarity of the stationary phase.

2.4 VOC - acronym for the volatile organic compounds.

2.5 Internal Standard - An analyte of known concentration added to standards, blanks and samples to measure the relative responses of target analytes. Internal standard concentration remains at a fixed value and acts as a reference point for system variances.

2.6 Surrogate Standard - A standard compound unlikely to be found in samples, but with similar chemical characteristics to target analytes. Known concentrations of these compounds are added to each sample and used to monitor method performance.

2.7 Laboratory Reagent And Method Blank - An aliquot of laboratory reagent water that is treated and analyzed in the same manner as analytical samples. This can also serve as the method blank for analysis of volatiles.

2.8 Laboratory Control Sample (LCS) - A laboratory control sample is a control matrix spiked with analytes representative of the target analytes in the method or a certified reference standard. The purpose of these samples is to determine method precision and monitor method control exclusive of sample matrix effects.

2.9 **Matrix Spike (MS)** - An aliquot of an analytical sample to which known quantities of target analytes have been added.

2.10 Matrix Spike Duplicate (MSD) - A duplicate aliquot of an analytical sample to which known quantities of target analytes have been added.
2.11 Neat Standards - Undiluted compound of at least 96% purity used to prepare stock standard solutions.
2.12 Stock Standard Solution - A concentrated solution of one or more compounds in solvent at a specified concentration. Stock standards are used to prepare primary dilution standards.
2.13 Primary Dilution Standards - A diluted mixed solution of compounds made from stock standards that is used to prepare calibration standards.
2.14 Working Calibration Standards - Prepared from the primary dilution of the stock standard, working calibration standards are used to calibrate the instrument response relative to analyte concentration.
2.15 Quality Control Sample (QCS) - A sample matrix or solution of analytes in a water miscible solvent used to fortify reagent water or environmental samples. The QCS is obtained from an external source and used to check laboratory performance.

3.0 REAGENTS

3.1 Purge and trap grade methanol , Burdick and Jackson cat.# 232-235.
3.2 Organic free reagent water, charcoal filtered or equivalent.
3.3 Hydrochloric acid : water , 1:1. Baxter catalog # 5587-6NY
3.4 Sodium thiosulfate ($Na_2S_2O_3$), reagent grade 99%. Aldrich catalog # 21,726-3 or equivalent.
3.5 Standards–Mixes 2000 ug/mL each.

- **3.5.1** Accustandard volatile liquids, cat. no. M-502A-R-10X.
- **3.5.2** Accustandard volatile gases, cat. no. M-502B-10X.
- **3.5.3** Accustandard 8260A compounds, cat. no. M-8260A-ADD-10X.
- **3.5.4** Neat Standards
  - **3.5.4.1** Toluene-d8, Aldrich # 26,985-9.
  - **3.5.4.2** 1,2-Dichloroethane-d4, Chem Service # F836.
  - **3.5.4.3** Dibromofluoromethane, Chem Service #F953.
  - **3.5.4.4** 1-Bromo-2-fluorobenzene, Aldrich # B6,680-9.
  - **3.5.4.5** 1-Bromo-4-fluorobenzene, Aldrich # B6,720-1.
  - **3.5.4.6** Fluorobenzene, Aldrich # F600-1.
  - **3.5.4.7** Pentafluorobenzene, Aldrich # P530-1.
  - **3.5.4.8** 1,4-Difluorobenzene, Aldrich # D10,220-2.
  - **3.5.4.9** Chlorobenzene-d5, Aldrich # 17,660-5.
  - **3.5.4.10** 1,2-Dichlorobenzene-d4, Aldrich # 2199-69-1.
  - **3.5.4.11** Chlorobenzene, Aldrich # 28,457-3.
  - **3.5.4.12** Toluene, Aldrich # 27,037-7.
  - **3.5.4.13** Benzene, Aldrich # 27,070-9.
  - **3.5.4.14** Trichloroethene, Aldrich # 25,642-0.
  - **3.5.4.15** 1,1-Dichloroethene, Aldrich # 16,302-3.

note: equivalent standards can be used when needed.

4.0 SUPPLIES

4.1 40 mL VOA vials with Teflon coated septa.

4.2 5 and 25 mL gas tight syringes.

4.3 Teflon coated stirbars for Archon autosampler.

4.4 10, 50, 100 and 1000 µL syringes for internal standards, surrogate standards and target compound calibration.

4.5 1, 8 and 40 mL vials w/ Teflon lined screw caps for storage of calibration standards.

4.6 5, 10, 50, and 100 mL Class A volumetric flasks.

5.0 INSTRUMENTS

5.1 *Gas Chromatograph*

5.1.1 Varian 3400 w/ 1077 split /splitless inlet system.

Column - J&W DB-624 60 meter x 0.320 mm ID fused silica with 1.8 um film thickness, in conjunction with a J&W DB-WAX column (30 meter x 0.53 with a 1.0 µm film) as a transfer line.

Flow rate - 1.0 mL/minute.
GC Method File - 8240
Inlet Temp.: 220 C
Open Split Interface: 250 C
Transfer Line: 250 C
Column Temperature Profile:
Initial: 45 C for 1 minute
Ramp 1: 7 C/min to 160 C
Ramp 2: 18 C/min to 240 C
Hold for 5.14 minutes.
Total run time: 27:00 minutes.

5.2 Purge and Trap Introduction System

5.2.1 Tekmar LSC 2000 purge and trap unit with moisture control module
Trap - Supelco Vocarb 3000

5.2.1.1 Water method - #1
Purge time - 11 min
Dry purge - 1 minutes
Desorb preheat - 245 C
Desorb - 250 degrees
Desorb time - 2 minute
Bake - 270 degrees
Bake time - 7:00 min

5.2.2 Varian Archon Purge & Trap Autosampler

5.2.2.1 Water samples : 5 mls
Purge flow - 40 mls/min

5.2.2.2 Soil samples: 5.0 g
Purge flow - 40 mls/min
Transfer line - 150 C
Sample heat - 40 C
Pre-heat - 3.0 min
Water volume - 10 mls
Pre-purge - 0.0 min
Purge time - 11 min
Soil stir - yes
Flushes - 2
Desorb time - 2 min
Trap volume - 0 mls

5.3 Mass Spectrometers

Aquire file - 8240		
Range - 35-260 m/z	Rate - 0.75 sec/scan	Filament on - 3.7 min
AGC target - 7700	Emission current - 11 uamps	EM voltage - 1850
AGC - on	Mass defect - 0	Background mass - 33 m/z

Varian Saturn GC/MS

- PURGE AND TRAP INTRODUCTION SYSTEM

Tekmar LSC 2000 purge and trap unit with moisture control module.
Tekmar 3000 Purge and Trap Concentrator
Varian Archon Purge and Trap Autosampler

Trap - Supelco Vocarb 3000 or Tenax/Silica gel/Charcoal

Water method - #1
Purge time - 11 min
Dry purge - 1 minutes
MCM Desorb temp - 0 degrees
Desorb preheat - 245
Desorb - 250 degrees
Desorb time - 2.00 minutes
Bake - 270 degrees
Bake time - 7.00 min
MCM bake - off

- MASS SPECTROMETER

Aquire file - WATER

Scan Range - 35 - 260 a.m.u.
Scan Rate - 0.75 sec/scan
Filament on - 4 min
AGC target - variable
Emission current - 11 uamps
EM voltage - variable
AGC - on
Mass defect - 0
Background mass - 33 a.m.u.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1 Samples stored in the laboratory are maintained at 4 degrees centigrade until analysis. Samples are kept in a solvent free refrigerator, separate from all standards, which is designated for volatiles samples only.
6.2 Unpreserved water samples must be analyzed within 7 days, and preserved waters and soils within 14 days, from the sampling date. If samples cannot be run within these time frames, the client will be notified.

7.0 PROCEDURES

7.1 Calibration standards must be made from neat material of at least 96% purity or purchased in diluted form with traceable standard purity. Stock solutions of internal standards are prepared from neat compounds as follows:

7.1.1 Measure out 8 mL of purge and trap grade methanol in a 9 mL septum sealed vial. Then tare the vial and methanol on the analytical balance.
7.1.2 Measure out enough compound to weigh approximately 50,000 ug using a 100 uL gas tight syringe. This can be estimated using the compound's density as follows:
bromofluorobenzene d = 1.593g/mL, 1.593 g/mL = 1,593 µg/uL 50,000 µg x 1/1,593 µg/mL = 31.4 µL of neat compound.
7.1.3 Add the compound to the tared vial and weigh to the nearest 0.0001g. Record the weight and retare the vial for the next compound.
7.1.4 Repeat this process until all standards are weighed and recorded.
7.1.5 After all standards have been weighed, quantitatively transfer the standards to a 10 mL class "A" volumetric flask and bring to volume with purge and trap grade methanol. This concentration (approximately 2000 µg/mL) will serve as a stock solution for calibrations, surrogate standards and internal standards.
7.1.6 Stock solutions of internal standards must be prepared separately, at a concentration of 5000 µg/mL.
7.1.7 Stock solutions of surrogate and target compounds are purchased mixed from Accustandard, Crescent Chemical, Ultra Scientific, Restek, or Supelco. Stock concentrations should be 2,000 µg/mL.
7.1.8 Working compound standards are prepared by diluting the stock solutions made above or by diluting prepared standards using the following scheme: Working internal standard/surrogate mix solution is prepared by adding 1.0 mL of stock internal standard solution and 2.5 mL of surrogate stock solution to a 25 mL volumetric flask and diluted to volume with reagent grade methanol. 1.0 µL (autosampler loop size) = 40 µg/L internal standard and 40 µg/L of surrogate standard for a 5mL or 5 gram sample. Prepare working standard at 200 µg/mL by diluting 2000µg/µL standard in a 10 ml volumetric. Store dilutions in autosampler vials.
7.1.9 Stock solutions and working standards are kept at <-10 C in the standards refrigerator in the Volatiles lab. All stock standards expire 1 year from date of preparation or the date listed by the providing manufacturer, whichever is sooner. All working standards expire 6 months from preparation or the date of the stock standard expiration, whichever is sooner.
7.1.10 Calibration curves are assembled using measured amounts of the working standards to 50 mL aliquots of reagent water as follows: 10 µL of 200 µg/mL in 50 mls = 40 µg/L. Typical concentrations for the working standards are 4, 8, 20, 40, 100, 200, 320, 500, and 1000 µg/l.
7.1.11 A minimum of 5 points are required for calibration for organic compounds. You may select to use all the standards above, more than those listed above or less than those above as long as a minimum of five

points are used and the successive concentrations differ by no more than one decade.

7.1.12 Tabulate the area response of the characteristic ions (see Section 1) against concentration for each compound and each internal standard. Calculate relative response factors (RRF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RRF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RRF is calculated as follows:

$RRF = (A_x)(C_{is})/(A_{is})(C_x)$ where:

A_x = Area of the characteristic ion for the compound being measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

7.1.13 The average RRF must be calculated and recorded for each compound. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average relative response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane; and chlorobenzene. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.1.13.1 Chloromethane - This compound is the most likely compound to be lost if the purge flow is too fast.

7.1.13.2 Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response. Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.1.14 Using the RRFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all compounds. The percent RSD is calculated as follows:

$$\% \text{ RSD} = \frac{SD}{RF_X} \times 100\%$$

Where: RSD = Relative standard deviation.

RF_X = mean of 5 initial RRFs for a compound.

SD = standard deviation of average RRFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{n} \frac{(RF_i - \overline{RF})^2}{n-1}}$$

Where: RF_i = RF for each of the 5 calibration levels.

n = number of RF values.

The percent relative standard deviation should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) must be less than 30%. The CCCs are:

1,1-Dichloroethene,
Chloroform,
1,2-Dichloropropane,
Toluene,
Ethylbenzene, and
Vinyl chloride.

If a %RSD greater than 30 percent is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is required before re-attempting calibration.

7.1.15 Linearity - If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. If the %RSD of any compound is greater than 15% construct calibration curves of area ratio (A/A(is)) versus concentration using first or higher order regression fit of the five calibration points.

7.2 GC/MS calibration verification

7.2.1 Prior to the analysis of samples, inject or purge 5-50 ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Section 8.5 before sample analysis begins. These criteria must be demonstrated each 12-hour shift.

7.2.2 The initial calibration curve (Section 7.1) for each compound of interest must be checked and verified once every 12 hours during analysis with the introduction technique used for samples. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC and CCC.

7.2.3 System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum relative response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.2.3.1 The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.1
1,1-Dichloroethane	0.1
Bromoform	> 0.1
Chlorobenzene	0.3
1,1,2,2-Tetrachloroethane	0.3

7.2.4 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs listed in Section 7.1.13 are used to check the validity of the initial calibration.

Calculate the percent drift using the following equation:

$$\% \text{ Drift} = (C(I) - C(c))/C(I) \times 100$$

where: C(I)= Calibration Check Compound standard concentration.

C(c) = Measured concentration using selected quantitation method.

If the percent drift for each CCC analyte is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% drift), for any one CCC analyte, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five point calibration MUST be generated. This criterion MUST be met before quantitative sample analysis begins. If CCC compounds are not required analytes by the permit, then all required analytes must meet the 20% drift criterion.

7.2.5 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two for any sample, blank or standard (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, all samples analyzed during the malfunction must be re-analyzed.

7.2.6 For compounds which exhibit linearity of response, the RRF of the daily check standard may be used for quantitation, provided the criteria for SPCCs and CCCs are satisfied.

7.3 Instrument Maintenance for Failing Verification (if sensitivity changes significantly a new MDL determination may be required.)

7.3.1 Check and adjust GC and/or MS operating parameters. Including, but not limited to carrier flow rate, mass calibration, electron multiplier voltage and target values.

7.3.2 Clean, silanize or replace injection port liner.

7.3.3 Remove a short section (1 meter) of column at the inlet end, backflush or replace the analytical column.

7.3.4 Prepare fresh calibration standards and repeat initial calibration.

7.3.5 Clean the mass spectral analyzer according to manufacturers specifications.

7.3.6 Replace any parts of the system that have become reactive to target analytes (transfer lines, valves, etc.,...).

7.3.7 Replace electron multiplier.

7.4 Qualitative Identification

7.4.1 Compounds are identified both by retention time and comparison of the sample mass spectrum, after background subtraction, with characteristic ions in a reference mass spectrum. Characteristic ions are either the three ions of greatest intensity or any ions over 30% relative intensity if less than three such ions exist. Compounds are identified when the following criteria occur:

7.4.1.1 The intensities of the characteristic ions of an analyte maximize within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target analyte at the analyte specific retention time is accepted as meeting this criterion.

7.4.1.2 The relative retention time, RRT, of the compound agrees within 0.06 RRT units to that of the standard target analyte.

7.4.1.3 The relative intensities of the characteristic ions agree within 30% to those found in the reference spectra. A reference ion at 50% relative intensity would require that the same sample ion be between 20% and 80% relative intensity for an acceptable match.

7.4.1.4 Some structural isomers have very similar spectra and should be identified by compound specific retention times where possible. Acceptable peak resolution for retention time identification is possible if the valley between two isomeric peaks is less than 25% of the sum of the two peak heights. When the valley between the two peaks is greater than 25% the peaks will be identified as isomeric pairs.

7.4.1.5 Complex sample matrices will be encountered that contain multiple components that are not completely chromatographically resolved. These samples may produce spectra that contain ions from several analytes, these may sometimes be identified as broadened peaks or peak shoulders. In these instances it is important to select the background subtraction carefully to produce a suitable spectral match. Extracted ion current profiles can be very beneficial in selecting a background subtraction point for closely eluting compounds. In the event that no chromatographic resolution exists, spectral identification using only the characteristic ions for the target compound is acceptable. Identification requires expert judgment when sample components are not chromatographically resolved. Spectra can contain ions from both analytes and must be interpreted correctly. A library search algorithm that uses "fit" criteria may be used or various automatic or manual subtraction techniques may be used to assist in compound identification.

7.4.2 It may become necessary to identify compounds that are not accounted for in the calibration standards. Tentative identification may be achieved by using an automated library search algorithm provided that the following guidelines are observed.

7.4.2.1 The relative intensities of major ions in the reference spectrum are present in the sample spectrum.

7.4.2.2 The intensities of the major ions are within $\pm$ 20% of the reference spectrum.

7.4.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

7.4.2.4 Ions present in the sample spectrum but not in the reference spectrum should be evaluated for possible sources such as background or peak coelutions.

7.4.2.5 Ions present in the reference spectrum but not in the sample spectrum should be evaluated for poor background subtraction, peak coelution or data system anomalies.

8.0 QUALITY ASSURANCE / QUALITY CONTROL

8.1 Syringes are calibrated once per month and recorded in a pipette calibration log. Syringes must be within ± 2% of the expected value.
8.2 Balance calibration is checked once per day using a NIST class "S" weight and recorded in a balance calibration log. Balances must be within ± 2% of the expected value.
8.3 The GC/MS system must be tuned by injecting or purging 10 ng of 4-bromofluorobenzene and demonstrating the system conforms to the following criteria on a 12 hour clock that begins with the injection of the tuning compound :

MASS (m/e)	Ion Abundance Criteria
50	15 - 40% of the base peak
75	30 - 60% of the base peak
95	Base peak, 100% relative abundance
96	5 - 9 % of the base peak
173	less than 2% of the base peak
174	greater than 50% of the base peak
175	5 - 9% of mass 174
176	greater than 95%, but less than 101% of mass 174
177	5 - 9% of mass 176

8.4 A system blank is prepared by adding 1.0 µL of internal standard/surrogate standard mix to 5 mL (or 25 mL) of reagent water and loaded into an appropriate sparging vessel. Run method #1 on the purge and trap (LSC 2000) for water. If the system blank shows no compounds above the method detection limit, you may proceed to running the daily calibration check sample. If compounds are found above the detection limit note it in the corrective actions log, identify and correct the problem. Methylene chloride is a common laboratory contaminant and must be > 5 times the stated detection limit to be reported. Prepare a new system blank and rerun. Repeat the process above until the system blank is free of contaminants above the detection limit.

> **8.4.1** A separate system blank for high level samples containing the same amount of methanol is required. This may be substituted for the system blank above provided that all criteria are met.

8.5 A calibration check sample is prepared by adding 1.0 uL of the internal standard/surrogate standard and 10 uL of a 200 µg/mL working calibration standard solution to a clean Voa filled with reagent water and purging a 5 ml aliquot. The actual mean volume of supplied VOAs is 42.9 ml, giving a concentration in the 5 ml aliquot of 46.5 ug/L. Calibration check samples are run every 12 hours and must contain all target analytes. The calibration check must meet the criteria for CCC's and SPCC's stated in Table 5 for sample analyses to proceed. All analytes in the calibration check sample must meet the 20 % drift criteria if they are included in the scope of work or project plan. In addition, the responses for the internal standards (pentafluorbenzene, 1,4-difluorobenzene, chlorobenzene-d5, and 1,2-dichlorobenzene-d4) must be within -50% to +100% of the last daily calibration check. If the calibration check sample passes the acceptance criteria, sample analysis may proceed. If the calibration check fails any of the acceptance criteria, note the incident in the corrective actions log, identify and correct the problem before proceeding. Prepare a new calibration check and rerun. Repeat the process above until all acceptance criteria are met.

8.6 Batch QC

8.6.1 A laboratory control sample(LCS), matrix spike(MS), and matrix spike duplicate(MSD) is run at a frequency of once per 20 samples for each matrix (Soil, Water, High Level Soil), or once per 30 days, whichever is more frequent. The spike concentration is 46.5 µg/L for water, 40 µg/Kg for soils, 5000 µg/Kg for high level soils.

8.6.1.1 Calculate the spike recoveries for the LCS, MS and MSD. If all recoveries are within the established limits in Table 3, The method is presumed in control and sample analysis can proceed.

8.6.1.2 If the spike recoveries for the LCS are within the established control limits in Table 3, but the MS(and/or MSD) are not within the established limits in Table 4, the method is presumed in control and sample analysis can proceed. Sample data for the spiked sample with recoveries outside of the acceptance limits in Table 3. should be flagged as "estimated concentration."

8.6.1.3 If the spike recoveries for the LCS are not within the established control limits in Table 3, corrective actions must be performed to bring the method back into control. After corrective actions are performed, the analyst(s) must demonstrate LCS recoveries within the established limits before sample analysis can proceed.

8.7 The compounds and criteria in Table 2. will be used to monitor surrogate compounds. Any surrogate compound that fails these criteria must be noted on the surrogate recovery form. Typically three surrogates are used for this method, surrogate recovery is considered out of control only if any surrogate fail to meet control limit specifications. Samples containing large amounts of components may obscure or enhance the surrogates and force the recovery outside of expected control limits. Samples can be diluted and rerun to determine accurate recoveries if necessary. If the sample is respiked with surrogates and rerun with the same or similar outlying results then the sample will be reported as having demonstrated matrix effects and that results reported should be interpreted with caution.

8.8 Corrective Actions

All out of control situations need to be documented, corrected and initialed by a quality assurance officer before proceeding with sample analyses. Once a situation is out of control it is not necessary to document every step taken to correct the problem, only a brief statement on how the problem was corrected. However; all corrective maintenance must be recorded in the instrument maintenance manual, analysis logbook, and corrective action form.

Failure of the criteria for initial calibration, calibration check, internal standard response or surrogate standard response will require one or all of the following:

8.8.1 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within laboratory established limits in table 2. If surrogate recovery is not within limits, the following procedures are required.

8.8.1.1 Check to be sure there are no errors in calculations, surrogate solutions. Also, check instrument performance.

8.8.1.2 Recalculate the data and/or re analyze the extract if any of the above checks reveal a problem.

8.8.1.3 Re extract and re analyze the sample, if none of the above are a problem or flag the data as "estimated concentration."

8.8.1.4 Laboratory control samples and preparation blanks must have surrogate recoveries within laboratory established limits.

8.8.2 If the laboratory control sample (LCS) is out of control the following procedures are required.

8.8.2.1 Check to be sure there are no errors in calculations, spiking solutions. Also, check instrument performance.
8.8.2.2 Recalculate the data and/or re analyze the extract if any of the above checks reveal a problem.
8.4.2.3 Re-analyze the LCS sample to demonstrate that the analysis is in control.
8.4.2.4 Re-extract and re-analyze the LCS and all samples associated with the unacceptable LCS.

8.8.3 Samples that are prepared and run with an out of control preparation blank must be re-extracted and re-run along with a new preparation blank.
8.8.4 Flag data from samples that have unacceptable matrix spike and/or matrix spike duplicate recoveries or precision outliers as "estimated concentration".

9.0 SAMPLE PREPARATION

9.1 Water samples - samples in 40 mL VOA vials can be loaded onto the autosampler tray without any additional preparation. The autosampler is capable of making automated, programmed dilutions up to 1:100. The autosampler will remove 5 mLs, add the internal standard and surrogates, and inject the sample into the sparging vessel to purge. The internal standards and surrogates are contained in a syringe with a 1.0 µl loop. The concentration in the syringe is 200 µg/mL, giving a final concentration of 40 µg/L: (200 µg/mL x 1.0 µL) / (5 mL)=40 µg/L.
9.2 Soil Samples - samples are weighed directly into soil sparing vessels on a 3 place electronic balance. Sample size is usually 5.00g +/- 0.01g, but smaller amounts can be used if high concentrations are suspected (using smaller sample sizes raises detection levels). A stir bar is added. Soil sparging vessels are then placed on the autosampler tray, which adds the internal standard and surrogates, water, heats and magnetically stirs the sample, and purges the sample. The internal standard and surrogates have a final concentration of 40 µg/kg.
9.3 High Level Soil -Four (4.0) gram sample aliquots are weighed directly into 20 mL disposable scintillation vials on a 3 place electronic balance and recorded to the nearest 0.01 gram. After weighing add 4µL of stock standard surrogate mix (5,000 µg, mL) and 10mL of purge and trap grade methanol. Cap and shake the sample vigorously for 10 minutes. After allowing the sediment to settle, add 100µL of the methanolic extract to 5 mLs of organic free water and purge using the soil method. Alternatively, a 1 mL aliquot of the extract may be added to a 50 mL volumetric flask, mixed and diluted to volume with organic free water. A 40 mL VOA vial is then filled to capacity and capped with the septum sealed cap. The VOA vial is then placed in the autosampler and run using the water method. If any of the contaminants present are still outside the calibration curve of the instrument or if sample screening suggests that levels are very high a smaller methanolic aliquot may be used.
9.4 Method blanks are made using the following procedures and processed using the sample prep procedures above for the corresponding matrix type:
9.4.1 A water blank is prepared by loading a 40 mL VOA filled with reagent water and capped with a septum sealed cap onto the autosampler and running method #1 for water on the autosampler. The autosampler will withdraw 5 mL of water, add 1 uL of internal standards and surrogates, and inject the water blank into the sparging vessel to purge.
9.4.2 A low level soil blank is prepared by weighing 5 grams (to the nearest 0.01 g) of clean lab sand into a clean 40 mL VOA vial, capping

with a cap and teflon lined septum seal. Place the vial into the autosampler tray and run method #2 for soil. The autosampler will add reagent water, internal standards and surrogates and purge the blank.

9.4.3 A high level blank is prepared by placing 4 grams (to the nearest 0.01 g) of clean lab sand and 10 mL of purge and trap grade methanol in a 20 mL disposable scintillation vial. Add 4 uL of stock standard surrogate mix. Add 100 uL of the extract to 5 mLs of reagent water in a 40 mL VOA and cap with a septum sealed cap. Purge under soil method on autosampler. Alternatively, a 1 mL aliquot of the extract may be added to a 50 mL volumetric flask, mixed and diluted to volume with organic free water. A 40 mL VOA vial is then filled to capacity and capped with the septum sealed cap. The VOA vial is then placed in the autosampler and run using the water method.

9.5 Laboratory control samples, matrix spikes and matrix spike duplicates are made using the following procedures and processed using the sample prep procedures above for the corresponding matrix type:

9.5.1 Water spikes are prepared by filling a 40 mL VOA with reagent water and capping with a septum lined cap. 10 uL of a 200 ug/mL working compound standard is then added directly through the septa. LCS is placed in autosampler tray and run under water method. For MS/MSD 2 separate aliquots of a single sample are similarly spiked and purged.

9.5.2 Low level soil spikes are prepared by adding 5.00 grams clean lab sand to a clean tared VOA along with 5 mL reagent water. 10 uL of 20 ug/mL working compound standard is added and the VOA capped with a septum sealed cap. For MS/MSD two separate aliquots of a single sample are similarly weighed and spiked. The VOA is placed on the autosampler and is run using the soil method.

9.5.3 High level soil spikes are prepared by adding 4.00 grams clean lab sand to a tared disposable scintillation vial, along with 10 mL purge and trap grade methanol. 4 uL stock standard mix is added along with 100 uL 200 ug/ml working compound standard. For MS/MSD two separate aliquots of the same sample are similarly weighed and spiked. Add 100 uL of the extract to 5 mLs of reagent water in a 40 mL VOA and cap with a septum sealed cap. Purge under soil method on autosampler. Alternatively, a 1 mL aliquot of the extract may be added to a 50 mL volumetric flask, mixed and diluted to volume with organic free water. A 40 mL VOA vial is then filled to capacity and capped with the septum sealed cap. The VOA vial is then placed in the autosampler and run using the water method.

10.0 CALCULATIONS

10.1 The sample amounts can be read from the calibration curve, but are usually calculated by the workstation software directly from the curve and reported in the appropriate units.

10.2 Multiply the computed concentration value by any dilution factor used. The workstation software provides a place for this factor to be added into the calculated value at the time of sequence setup.

10.3 If the dry weight values are to be reported divide the value from above by the percent solids value obtain from that calculation.

10.4 The equation for sample concentration is as follows:

$X = C \times D$ (for wet weight determinations)

$X = (C \times D/S) \times 100$ (for dry weight calculations)

where :

X = sample concentration C = calculated concentration

D = dilution factor

S = Percent solids calculation = $\frac{\text{dry weight of sample}}{\text{wet weight of sample}} \times 100$

10.5 The equation for sample spike and spike duplicate recovery is as follows:

$$R = (X_k - X_s)/K \times 100\ \%$$

where :

R = spike recovery X_k = spike concentration

X_s = sample concentration K = amount spiked

10.6 The equation for relative percent difference is as follows:

$$RPD = | R - R' |/((R + R')/2) \times 100\%$$

where :

RPD = relative percent difference

R = spike recovery R' = spike duplicate recovery

10.7 The equation for calculating concentration through the software is as follows:

$$\text{For water: concentration (ug/L)} = \frac{(A_x)\ (I_s)}{(A_{is})\ (RRF)\ (V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured.

I_s = Amount of internal standard injected (ng).

A_{is} = Area of characteristic ion for the internal standard.

RRF = Relative Response factor for compound being measured.

V_o = Volume of water purged (mL), taking into consideration any dilutions made.

Sediment/Soil Sludge and Waste(normally on a wet-weight basis)

$$\text{concentration (ug/kg)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_i)(W_s)(D_s)}$$

where:

A_x = Area of characteristic ion for compound being measured.
I_s = Amount of internal standard injected (ng).
A_{is} = Area of characteristic ion for the internal standard.
RRF = Relative Response factor for compound being measured
V_t = Volume of total extract (uL) (use 10,000 uL or a factor of this when dilutions are made).
V_i = Volume of extract added (uL) for purging.
W_s = Weight of sample extracted or purged (g).
D_s = 1 for a wet-weight, or from section 10.4 above which is:

$$S/100 = \frac{\text{dry weight of sample}}{\text{wet weight of sample}}$$

High level soils prepped by methanolic extraction:

$$\text{concentration (ug/kg)} = \frac{(A_x)\ (C_{is})\ (V_t)}{(A_{is})\ (RRF)\ (V_i)\ (W_s)}$$

where :

A_x = area of the characteristic ion of the analyte being measured
A_{is} = area of the characteristic ion of the specific internal standard
C_{is} = concentration of the specific internal standard in nanograms (ng).
RRF = relative response factor
V_t = total volume of methanolic extract in uL
V_i = volume of extract purged for analysis in uL
W_s = weight of sample aliquot extracted

11.0 METHOD SUMMARY

11.1 All sample results, chromatograms and data sheets must be stored with the sample chain of custody in the client file. All instrument maintenance, initial calibration, continuing calibration, tuning data, spike, duplicate spike, blank and corrective actions data are recorded in the instrument log book. All pipette calibration, analytical balance calibration, percent solids, sample preparation and associated quality assurance data should be recorded in the appropriate log books. Raw data for quality control samples is stored in the QC file cabinet in chronological order. Mass spectral data is stored onto magnetic storage tapes and archived for a period of five years (ten years for VAP data). These tapes are to be stored in a fire safe in Suite A-8. Hard copy data files are kept for a period of five years (ten years for VAP data).

12.0 REFERENCES

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12.2 APHA, AWWA, WPCF, 1986, "Standard Methods For The Examination Of Water And Wastewater" , 16th Edition, American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005.

12.3 Association of Official Analytical Chemists, 1980, " Official Methods Of Analysis" . 14th Edition, Association of Official Analytical Chemists, P.O. Box 540, Benjamin Franklin Station, Washington, DC 20044.

12.4 ASTM, "Annual Book Of ASTM Standards", 17th Edition,1989,American Society Of Testing And Materials, 1916 Race Street, Philadelphia, PA 19103.

12.5 U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1983, "Methods For Chemical Analysis Of Water And Wastes", Environmental Monitoring and Support Laboratory, Office of Research and Development. Cincinnati,OH 45268.

12.6 U.S. Environmental Protection Agency, November 1986, Third Edition, "Test Methods For Evaluating Solid Waste", Office of Solid Waste and Emergency Response, Washington, DC 20460.

12.7 Federal Register, July 8th, 1987, "National Primary Drinking Water Regulations - Synthetic Organic Chemicals; Monitoring For Unregulated Contaminants; Final Rule." Vol. 52, 130, 25690 -25718.

12.8 Federal Register, March 29, 1990, "Hazardous Waste Management: Identification And Listing Of Hazardous Waste; Toxicity Characteristic Revision; Final Rule". Vol. 55, No. 61, 1798 - 1877.

APPENDIX A

GC/MS VOLATILES TABLES

TABLE 1.
EPA METHOD 8260A
QC Acceptance Criteria and Detection Limit Summary

COMPOUNDS	Accuracy	Precision	Completeness	Water PQL (ug/L)	Soil PQL (ug/kg)	Water MDL (ug/L)	Soil MDL (ug/kg)
Dichlorodifluoromethane	40 - 160	0 - 30	95	5.0	5.0	0.3633	0.1043
Chloromethane	D - 273	0 - 30	95	5.0	5.0	0.3048	0.1349
Vinyl chloride	D - 251	0 - 30	95	5.0	5.0	0.3088	0.1236
Bromomethane	D - 242	0 - 30	95	5.0	5.0	0.2131	0.1272
Chloroethane	14 - 230	0 - 30	95	5.0	5.0	0.2729	0.1973
Trichlorofluoromethane	17 - 181	0 - 30	95	5.0	5.0	0.2494	0.0663
1,1-Dichloroethene	D - 234	0 - 30	95	5.0	5.0	0.3083	0.1505
Acetone	40 - 160	0 - 30	95	25.0	25.0	5.8931	6.6938
Carbon disulfide	40 - 160	0 - 30	95	5.0	5.0	0.2948	0.1730
MTBE	40 - 160	0 - 20	95	5.0	5.0	0.5551	0.2280
Methylene chloride	D - 221	0 - 30	95	5.0	5.0	0.1830	0.1369
2-Butanone	40 - 160	0 - 30	95	25.0	25.0	2.9761	2.4770
cis-1,2-Dichloroethene	40 - 160	0 - 30	95	5.0	5.0	0.1510	0.1424
trans-1,2-Dichloroethene	54 - 156	0 - 30	95	5.0	5.0	0.1094	0.1600
1,1-Dichloroethane	59 - 155	0 - 30	95	5.0	5.0	0.2680	0.1894
Chloroform	51 - 138	0 - 30	95	5.0	5.0	0.2799	0.1659
1,1,1-Trichloroethane	52 - 162	0 - 30	95	5.0	5.0	0.1961	0.1291
Carbon tetrachloride	70 - 140	0 - 30	95	5.0	5.0	0.1460	0.1227
Benzene	37 - 151	0 - 30	95	5.0	5.0	0.1894	0.1249
1,2-Dichloroethane	49 - 155	0 - 30	95	5.0	5.0	0.2149	0.1806
Trichloroethene	71 - 157	0 - 30	95	5.0	5.0	0.1498	0.1144
1,2-Dichloropropane	D - 210	0 - 30	95	5.0	5.0	0.2700	0.1696
Bromodichloromethane	35 - 155	0 - 30	95	5.0	5.0	0.1392	0.0691
cis-1,3-Dichloropropene	D - 227	0 - 30	95	5.0	5.0	0.1283	0.1470
4-Methyl-2-pentanone	40 - 160	0 - 30	95	25.0	25.0	2.2281	3.1779
Toluene	47 - 150	0 - 30	95	5.0	5.0	0.1131	0.1018
trans-1,3-Dichloropropene	17 - 183	0 - 30	95	5.0	5.0	0.2831	0.0997
1,1,2-Trichloroethane	52 - 150	0 - 30	95	5.0	5.0	0.3142	0.2235
2-Hexanone	40 - 16	0 - 30	95	25.0	25.0	2.1764	2.5559
Tetrachloroethene	64 - 148	0 - 30	95	5.0	5.0	0.1750	0.1369
Dibromochloromethane	53 - 149	0 - 30	95	5.0	5.0	0.0631	0.1337
Chlorobenzene	37 - 160	0 - 30	95	5.0	5.0	0.1717	0.1133
Ethylbenzene	37 - 162	0 - 30	95	5.0	5.0	0.2188	0.1519
Bromoform	45 - 169	0 - 30	95	5.0	5.0	0.2956	0.2500
1,1,2,2-Tetrachloroethane	46 - 157	0 - 30	95	5.0	5.0	0.4108	0.3269
1,3-Dichlorobenzene	59 - 156	0 - 30	95	5.0	5.0	0.3219	0.1268
1,4-Dichlorobenzene	18 - 190	0 - 30	95	5.0	5.0	0.2540	0.1299
1,2-Dichlorobenzene	18 - 190	0 - 30	95	5.0	5.0	0.1569	0.1955
m&p-Xylene	40 - 160	0 - 30	95	5.0	5.0	0.3497	0.2574
o-Xylene	40 - 160	0 - 30	95	5.0	5.0	0.1306	0.0854
Styrene	40 - 160	0 - 30	95	5.0	5.0	0.1518	0.0685
2,2-Dichloropropane	40 - 160	0 - 30	95	5.0	5.0	0.3708	0.1245
Bromochloromethane	40 - 160	0 - 30	95	5.0	5.0	0.1834	0.1606
Dibromomethane	40 - 160	0 - 30	95	5.0	5.0	0.0889	0.1964
1,3-Dichloropropane	40 - 160	0 - 30	95	5.0	5.0	0.3219	0.1057
1,2-Dibromoehtane	40 - 160	0 - 30	95	5.0	5.0	0.1928	0.1623
1,1,1,2-Tetrachloroethane	40 - 160	0 - 30	95	5.0	5.0	0.2215	0.1291
Isopropylbenzene	40 - 160	0 - 30	95	5.0	5.0	0.1213	0.0918
1,2,3-Trichloropropane	40 - 160	0 - 30	95	5.0	5.0	0.3973	0.3308
n-Propylbenzene	40 - 160	0 - 30	95	5.0	5.0	0.1851	0.0875
Bromobenzene	40 - 160	0 - 30	95	5.0	5.0	0.1840	0.2112
1,3,5-Trimethylbenzene	40 - 160	0 - 30	95	5.0	5.0	0.2070	0.1577
2-Chlorotoluene	40 - 160	0 - 30	95	5.0	5.0	0.2000	0.1336
4-Chlorotoluene	40 - 160	0 - 30	95	5.0	5.0	0.1918	0.1707
t-Butylbenzene	40 - 160	0 - 30	95	5.0	5.0	0.2359	0.1581
1,2,4-Trimethylbenzene	40 - 160	0 - 30	95	5.0	5.0	0.3405	0.1522
sec-Butylbenzene	40 - 160	0 - 30	95	5.0	5.0	0.1989	0.1996
4-Isopropyltoluene	40 - 160	0 - 30	95	5.0	5.0	0.1579	0.1467
n-Butylbenzene	40 - 160	0 - 30	95	5.0	5.0	0.1863	0.0838
1,2-Dibromo-3-chloropropane	40 - 160	0 - 30	95	5.0	5.0	0.6235	0.2889
1,2,4-Trichlorobenzene	40 - 160	0 - 30	95	5.0	5.0	0.2041	0.1712

Hexachlorobutadiene	40 - 160	0 - 30	95	5.0	5.0	0.6566	0.2371
Naphthalene	40 - 160	0 - 30	95	5.0	5.0	0.1851	0.1791
1,2,3-Trichlorobenzene	40 - 160	0 - 30	95	5.0	5.0	0.3973	0.3308
1,1-Dichloropropene	40 - 160	0 - 30	95	5.0	5.0	0.2412	0.1743

TABLE 2.
SURROGATE RECOVERIES FOR GC/MS METHODS

[illegible]	[illegible]	[illegible]	[illegible]
1,2-Dichloroethane d4	64-130	79-120	60-133
Toluene d8	62-132	76-114	58-124
Bromofluorobenzene	81-135	64-131	56-147

TABLE 3.
Acceptance Limits for Laboratory Control Samples on Volatile Organics by GC/MS

[illegible]	[illegible]	[illegible]	[illegible]
1,1-Dichloroethene	72-129	79-130	34-144
Trichloroethene	70-134	81-123	66-123
Benzene	77-127	82-123	57-127
Toluene	74-120	77-124	73-126
Chlorobenzene	77-125	78-125	62-126

TABLE 4.
Acceptance Limits for Matrix Spikes, and Spike Duplicates on Volatile Organics by GC/MS

[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
1,1-Dichloroethene	57-133	16	49-118	12	43-132	14
Trichloroethene	62-145	11	23-134	22	64-125	13
Benzene	62-142	11	44-115	21	58-131	9
Toluene	63-131	13	36-104	19	71-129	15
Chlorobenzene	68-124	12	31-109	21	56-133	10

TABLE 5.
Calibration Check Compounds (CCC)

CCC's for VOLATILE ORGANICS	Recommended %RSD for Linear Fit	Maximum %RSD for Initial Calibration	Maximum %Drift* for Continuing Calibration
1,1-Dichloroethene	15	30	20
Chloroform	15	30	20
1,2-Dichloropropane	15	30	20
Toluene	15	30	20
Ethylbenzene	15	30	20
Vinyl Chloride	15	30	20

System Performance Check Compounds (SPCC)

SPCC's for VOLATILE ORGANICS	Minimum RRF	Minimum RRF for Continuing Calibration
Chloromethane	0.10	0.10
1,1-Dichloroethane	0.10	0.10
Bromoform	> 0.10	> 0.10
Chlorobenzene	0.30	0.30
1,1,2,2-Tetrachloroethane	0.30	0.30

* - % Drift = $(C_1 - C_c)/C_1$ X 100 where:
C_1 = concentration of the CCC standard

C_c = measured concentration using selected quantitation method.

Initial calibration criteria for the remaining compounds states that if %RSD for the five point curve is less than or equal to 15%, the relative response factor is assumed to be constant over the calibration range and the average response factor may be used. If the %RSD is greater than 15%, a calibration curve is constructed using the area ratio (A/A_{is}) versus concentration using the first or higher order regression fit of the five point curve. The regression fit that introduces the least amount of error should be selected.

Attachment QAPP-B4

Analysis of Diesel Range Organics by Gas Chromatography
Method 8015 (DRO)

GAM 8015(DRO)

Analysis of Diesel Range Organics by Gas Chromatography w/FID

Revision 1.2 : 02/02/2002

INSTRUMENTATION: GC

1.0 SCOPE AND APPLICATION

ANALYTES:

1.1 This method is designed to measure the concentration of diesel range organics in water and soil. This corresponds to an alkane range of C10-C32 and a boiling point range between approximately 170-C and 430-C.

1.2 This method is designed to measure mid-range petroleum products such as diesel and fuel oil. Components greater than C-32 present in product such as motor oils or lubricating oils are detectable under the conditions of the method.

2.0 SUMMARY OF METHOD

2.1 GAM 8015(DRO) is based on a solvent extraction, GAM 3510B or GAM 3550A , Gas Chromatography procedure. One liter of water or 10 gram of soil is extracted with methylene chloride. Dilution may be performed as necessary to put the chromatographic envelope with in the linear range of the method. Quantitation limits are based on 1000 ug/mL of diesel in the extract and are 1.0 mg/L for waters and 4.0 mg/Kg for soils. A 2-uL aliquot of the extract is injected into a gas chromatograph (GC), and compounds in the GC effluent are detected by a flame ionization detector (FID).

3.0 INTERFERENCES

3.1 Other organic compounds including animal and vegetable oil and grease, chlorinated hydrocarbons, phenols and phthalate esters are measurable under the conditions of this method. As defined in the method, the DRO results include these compounds. Alumna column clean up may be used for the separation of sample extracts in to aliphatic aromatic and polar fractions.

3.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials just be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing method blanks.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak areas and/or peak heights is used. Hewlett Packard Chemstation 3365 is used to integrate and report data.

4.1.2 Columns:

Column 1: 30-m x 0.32-m I.D. 0.25 µm film thickness Restek RTX-5 fused silica capillary column. Column 2: 30-m x 0.32-m ID

1um film thickness Restek Rtx-200 fused silica capillary column

4.1.3 Detector: Flame ionization (FID).

4.2 Class A volumetric flasks: 10-, 50-, and 100-mL, with ground-glass stopper.

4.3 Microsyringe: 10-uL.

4.4 Scintillation Vials, 20 mL

4.5 Vials, 1.8 mL clear, autosampler with screw caps and Teflon coated septa.

5.0 REAGENTS

5.1 Solvents: Methylene chloride, Hexane, Acetone (pesticide quality or equivalent).

5.2 Stock standard solutions:

5.2.1 TPH standard is purchased from Restek as XHc diesel composite standard cat.# 31259, concentration 50,000 ug/mL. Serial dilutions in methylene chloride are made and detailed in Table 3.

5.2.2 Stock Continuing Calibration Check Standard is purchased from Restek(DRO mix cat# 31064), containing 10 components(Decane-Octacosane). See Table 2.

5.2.3 Dotriacontane(C32),97% Aldrich cat# D22,310-7. Purchased for retention time definition. C-10 to C-32 retention time range is 4.0-22.5 min.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Water and soil samples are stored refrigerated at 4°C. Water samples must be extracted within seven (7) days and soil samples extracted within 14 days of sample collection. Chemical addition 10% $Na_2S_2O_3$ to water samples is only recommended if residual chlorine is present.

6.2 Extracts must be stored under refrigeration and must be analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1- In general, water samples are extracted at a neutral pH with methylene chloride, using GAM 3510B. Solid samples are extracted with methylene chloride using GAM 3550A.

7.1.2 Table 2 in method GAM 3510B gives method specific data for spike standard concentration for water samples and table 3 in method GAM 3550A gives method specific data for spike standard concentration for soil samples. Method GAM 3510B shows appropriate final volume of 1 mL for GAM 8015 DRO in table 1. Method GAM 3550A gives appropriate final volume of 1 mL for GAM 8015 DRO in table 2.

7.2 Gas chromatography conditions (Recommended):

7.2.1 Gas chromatography conditions Column 1 and 2 :
Set helium carrier gas flow at 2-15-mL/min. flow rate. Set column temperature at 45-C for 1 min.; then program at 10-C/min to 100-C. 30-c/min to 190-C and finally 8-C/min to 290 and hold for 12 min. E.P.C is initialized at 25-psi for 1.0 min.; then program at 99 psi/min. to 22 psi

for 0 min. Temperature and pressure settings can be changed to optimize instrument performance.

7.3 Calibration

7.3.1 Calibration standards must be made from neat material of at least 96% purity or purchased in diluted form with traceable standard purity (i.e. Restek XHc diesel composite standard cat.# 31259, concentration 50,000 ug/mL). Stock solutions of calibration check standards and analyte compounds are prepared from neat compounds as follows:

7.3.2 Measure out 5 mL of methylene chloride in a 9 mL septum-sealed vial. Then tare the vial and solvent on the analytical balance.

7.3.3 For standards made from neat stock standards measure out enough compound to weigh approximately 50,000 µg using a 100 µL gas-tight syringe. This can be estimated using the compound density as follows:

Diesel fuel #2 d = 0.830 g/mL 0.830 g/mL = 830 µg/µL
50,000 µg/mL x 1µL/830 ug/µL = 60 µL of neat compound.
(60 µL x 830 µg/µL)/10 mL methanol ≈ 5,000 µg/ml

Add the compound to the tared vial and weigh to the nearest 0.0001g. Record the weight and re-tare the vial for the next compound. Repeat this process until all standards are weighed and recorded.

7.3.4 After all standards have been weighed or measured, quantitatively transfer the standards to a 10 mL class "A" volumetric flask and bring to volume with methylene chloride. The mix will serve as a stock solution for calibrations and calibration check standards and represent the total concentration of all compounds.

7.3.5 Prepare working standards by diluting aliquots of the 50,000 µg/ml stock standard described in table 2 to the following scheme:

1 mL of 50,000 µg/ml brought to 5 mL with solvent = 10,000 µg/ml
1 mL of 10,000 µg/ml brought to 5 mL with solvent = 2,000 µg/ml
1 mL of 2,000 µg/ml brought to 5 mL with solvent = 400 µg/ml
1 mL of 400 µg/ml brought to 5 mL with solvent = 80 µg/ml
1 mL of 80 µg/ml brought to 5 mL with solvent = 16 µg/ml
1 mL of 16 µg/ml brought to 5 mL with solvent = 3.2 µg/ml

7.3.6 Calibration curves are assembled using measured amounts of the working standards added to aliquots of reagent water as shown in table 2.

7.3.7 A minimum of 5 points are required for calibration for organic compounds. You may select to use all the standards above, more than those listed above or less than those above as long as a minimum of five points are used and the successive concentrations differ by no more than one decade. Average response factors can be used to calculate concentrations, if the RSD (relative standard deviation) is less than 20% over the calibrated range.

7.3.9 A continuing calibration check sample (made from, Restek(DRO mix cat# 31064) at the concentration stated in section 7.4.1) must be run daily to confirm the validity of the initial curve and must not exceed ±15% of the expected value. This calibration check sample is run at the

beginning, after every 10th sample and at the end of the sample sequence.

7.3.10 Calibration curves are constructed by plotting the total area response of the peaks against the concentration of the standard in total µg .

7.3.11 Diesel range organic concentrations are determined by multiplying the average calibration factor of the curve by the total area response from the sample.

A_{tot} X CF = C_S

where :
A_{tot} = the sum of all peak areas integrated between C10 - C32
CF = the calibration factor (calculated from 7.8.14.2)
C_S = the sample concentration in ug/sample.

7.3.12 The procedure for external standard calibration is used for gas chromatographic analyses. Calibration levels and individual components of the TPH standards are listed in Table 2.

7.3.13 Assemble a calibration curve by running the standards in 7.3.7 under the chromatographic conditions used for the method. Use the Chemstation software to assemble an external standard curve based on the integrated areas of the peaks of interest.

7.3.14 External standard calibration procedure

7.3.14.1 For each analyte of interest, prepare calibration standards at a minimum of five concentrations by adding volumes of one or more working standards to a volumetric flask and diluting to volume with organic solvent. One of the external standards should be at a concentration near, but above, the method reporting limit. The other concentrations should correspond to the working range of the detector.

7.3.14.2 Calculate the calibration factor (CF) by introducing each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. Tabulate total peak area responses against the mass injected. The results can be used to prepare a calibration curve for each analyte.

7.3.14.3 The working calibration curve or calibration factor must be verified on each working day by the injection of one or more calibration standards. If the response for any analyte varies from the predicted response by more than +/- 15%, a new calibration curve must be prepared for that analyte.

$$\text{Percent Difference} = \frac{R1 - R2 \times 100}{R1}$$

where:
R1 = Calibration Factor from first analysis.
R2 = Calibration Factor from succeeding analyses.

7.3.15 Load the file for the lowest standard run, integrate the file to return the peak areas and retention times of the peaks in the chromatogram. Select "Prep /Recalibrate" to create a calibration table and select "New" to make a new table. This function loads any integrated peaks into a calibration table. Use the cursor to select any unwanted peaks and use the "Remove Peak" key to remove peaks that are not desired in the calibration table. The "Add peak" key may be used to introduce a new peak (analyte) to an existing calibration table. Prepare the first calibration level and enter the appropriate amount in micrograms. Repeat this process for each successive standard until all the standard concentration levels are entered. Peaks that are manually integrated or substituted during analysis must be verified and initialed by the QA officer.

7.3.16 After the calibration file is complete, save the method and the calibration file will be saved with the method file. Select "overwrite" if the method file already exists.

7.4 Gas chromatographic analysis

7.4.1 Semi-volatile organics are usually introduced by direct injection. Follow the instructions on the analyses sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a 200 ug/sample calibration check standard (equivalent to a 200 mg/L water or a 6.65 mg/Kg soil (30g) or a 100 mg/Kg soil (2g) total TPH) at the beginning, after each group of 10 samples and at the end of the analysis sequence.

7.4.2 The appropriate detector is flame ionization detector.

7.4.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration check standards after every tenth (10^{th}) sample. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. Record the sample numbers and order run in the sequence logbook for each run sequence. The resulting peak sizes in area units are saved by the data system software and processed using an automated integration/calibration package.

7.4.4 Direct injection - inject the appropriate aliquot of the sample extract. Record the volume of the sample extract injected in the sample description to the nearest 0.01 µL and the resulting peak size in area units. Using the external calibration procedure, determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes. See Table 1 for calculation equations.

7.4.5 If the responses exceed the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

7.4.6 If peak detection is prevented by the presence of interferences, confirmation by a qualitative technique such as GC/MS may be necessary.

7.4.7 Examples of chromatograms for the compounds of interest from the calibration standards may be useful for comparing to sample chromatograms, especially when closely eluting components are present.

7.4.8 Calibrate the system immediately prior to conducting any analyses (see Section 7.3). A calibration check standard (7.4.1) must also be introduced after every ten analytical samples and at the end of the analysis sequence. The calibration factor for each analyte to be quantitated, must not exceed a 15% difference when compared to the initial standard of the analysis sequence. When this criteria is exceeded, inspect the GC system to determine the cause and perform whatever maintenance is necessary (see Section 7.5) before re-calibrating and proceeding with sample analysis. All samples that were purged after the standard exceeding the criteria must be re-purged, if the initial analysis indicated the presence of the specific target analytes that exceeded the criteria.

7.4.9 Establish daily retention time range for the fuel type. Use the absolute retention time for two components from Section 7.4.8 as the midpoint of the window for that day. Use the retention time of decane (C10)as a representative of the beginning of the carbon range and the retention time of dotriacontane (C32)as a representative of the end of the carbon range. The daily retention time window equals the midpoint +/- three times the standard deviation determined in Section 8.4.

7.4.9.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time range. Second column confirmation is not generally required for petroleum hydrocarbon analysis, profile pattern recognition using known standards is recommended for each fuel type. It is recommended that several fuel standards be chromatographed under the operating conditions of each column or oven program used so that a fuel profile is available for pattern matching when needed. At a minimum, chromatograms of fresh diesel, 50% weathered diesel, VM&P naphtha, Jet A, kerosene, mineral spirits and motor oil (10W30) should be run to provide profile patterns for comparison. Since the flame ionization detector is non-specific it is highly recommended that GC/MS confirmation be performed on single component analytes unless historical data is available to support the identification(s).

7.4.9.2 Validation of GC system qualitative performance: Use the calibration check standards interspersed throughout the analysis sequence (Section 7.4.8) to evaluate this criterion. If any of the standards fall outside their daily retention time window, the system is out of control. Determine the cause of the problem and correct it (see Section 7.5). All samples that were injected after the standard failing criteria must have a new sample aliquot injected to avoid false negatives and possibly false positives.

7.5 Suggested chromatography system maintenance - Corrective measures may require any one or more of the following remedial actions.

7.5.1 Capillary columns - Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert. Break off the first few inches, up to one foot, of the injection port side of the column. Remove the column and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the column.

7.5.2 Metal injector body - Turn off the oven and remove the analytical column when the oven has cooled. Remove the glass injection port insert (instruments with off-column injection or Grob). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

7.5.2.1 Place a beaker beneath the injector port inside the GC oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then toluene; catching the rinsate in the beaker.

7.5.2.2 Prepare a solution of deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, serially rinse the injector body with toluene, methanol, acetone, and hexane. Reassemble the injector and replace the GC column.

8.0 Quality Control

8.1 Preparation blanks, (laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent, for soils),(LCS and LCSD, for waters). LCS,LCSD, MS and MSD are made from Restek DRO mix cat# 31064 at a concentration 200 ug/sample DRO TPH.

8.1.1 Calculate the values for the preparation blanks, laboratory control samples (LCS), laboratory controlsample duplicates (LCSD),matrix spikes(MS) and matrix spike duplicates(MSD)

8.1.1.1 If the preparation blank shows no contamination above the reporting limits (Table 6) for the analytes of interest, the method is presumed in control and sample analysis can proceed.

8.1.1.2 If the preparation blank contains contamination above the reporting limit, corrective actions must be performed to bring the method back into control. After the corrective actions are performed the analyst(s) must demonstrate that the preparation and analysis procedures are free of contaminants before sample analysis can proceed.

8.1.1.3 Calculate the spike recoveries for the LCS, LCSD, MS and MSD. If all recoveries are within the established limits in tables 4 and 5. The method is presumed in control and sample analysis can proceed.

8.1.1.4 If the spike recoveries for the LCS are within the established control limits , but the MS (and/or MSD)are not within the established limits , the method is presumed in control and sample analysis can proceed. Sample data for the spiked sample with recoveries outside of the acceptance limits in table 5 should be flagged as "estimated concentration."

8.1.1.5 If the spike recoveries for the LCS are not within the established control limits in table 4, corrective actions must be performed to bring the method back into control. After corrective

actions are performed, the analyst(s) must demonstrate LCS recoveries within the established limits before sample analysis can proceed.

8.2 Gas Chromatographic System Check

8.2.1 The quality control check sample concentrate should contain each analyte at a concentration of 200 ug/sample in methylene chloride:

8.2.2 All compounds in the DRO range should be quantitated.

8.3 Retention time windows(also refer to GQAP 003)

8.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures and multi-response products (i.e. PCBs) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

8.3.2 Calculate the standard deviation of the three absolute retention times (use any function of retention time; including absolute retention time, or relative retention time) for each single component standard. For multi-response products, choose one major peak from the envelope and calculate the standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in samples.

8.3.2.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. For multi-response analytes (i.e. TPH), the analyst should use the retention time window, but should primarily rely on pattern recognition.

8.3.2.2 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

8.3.3 The analyst must calculate retention time windows for each analyte on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

8.4 Corrective Actions

8.4.1 If the laboratory control sample (LCS), is out of Quality control limits the following procedures are required.

8.4.1.1 Check to be sure there are no errors in calculations, spiking solutions. Also, check instrument performance.

8.4.1.2 Recalculate the data and/or re analyze the extract if any of the above checks reveal a problem.

8.4.1.3 Re-analyze the LCS sample to demonstrate that the analysis is in control.

8.4.1.4 Re-extract and re-analyze the LCS and all samples associated with the unacceptable LCS.

8.4.2 Samples that are prepared and run with an out of control preparation blank must be re-extracted and re-run along with a new preparation blank.

8.4.3 Flag all sample data for unacceptable matrix spike, matrix spike duplicate and RPD as " estimated concentration".

9.0 METHOD PERFORMANCE

9.1 Method performance data is presented in Tables 4 and 5.

9.2 The method detection limit for soil calculated according to 40 CFR, Part 136, Appendix B was 1.6 mg/Kg (external standard calibration method). A recommended practical quantitation limit is 4 mg/Kg for soil and 0.1 mg/L for water.

9.3 This method was tested by 13 laboratories Single operator precision, overall precision and method accuracy were determined.

10.0 REFERENCES

10.1 USEPA "SW-846 Test Methods for evaluating solid Waste," 3rd Edition, Method 8000,8100,3510,3550.

10.2 "Leaking Underground Fuel Tank(LUFT) field Manual, "State Water Resources Control Board, State of California, Sacramento, CA, May 1988.

10.3 Bellar, T.A., and J.J Lichtenberg, J. Amer. Water Works Assoc., 66(12), PP-739-7441974.

10.4 U.S. EPA 40 CFR Part 136,"Guidelines Establishing Test Procedures for the Analysis of pollutants Under the Clean Water Act; Final Rule and interim Final Rule and Proposed Rule," October26,1984.

TABLE 1.
Equations Used for Analytes by GC with Direct Injection External Standard Quantitation

Parameter	Equation	Units
Water concentration	$\frac{(A_x)(A)(V_t)}{(A_s)(V_i)(V_s)} \times d$ where : A_x = peak area of the of the analyte being measured. A = amount of standard injected in nanograms (ng). d = dilution factor, dimensionless. V_i = volume injected in µL A_s = peak area of the external standard. V_t = final volume of extract in mL. V_s = volume of sample extracted in mL.	µg/L
Soil concentration (wet weight)	$\frac{(A_x)(A)(V_t)}{(A_s)(V_i)(W_s)(D_s)} \times d$ where : A_x = peak area of the of the analyte being measured. A = amount of standard injected in nanograms (ng). d = dilution factor, dimensionless. V_i = volume injected in µL. A_s = peak area of the external standard. V_t = final volume of extract in uL. W_s = weight of sample extracted in grams. D_s = 1 for wet weight, or S/100 where : S = % solids = $\frac{\text{dry weight of sampl}}{\text{wet weight of samp.}} \times 100$	µg/Kg

Table 2.
Stock Standard Concentrations for Calibration Checks, Matrix Spikes, Control Samples and GC Performance Checks D22,310-7

Decane	D90-1	31064	4.59	20	20
Dodecane	29,787-9	31064	7.27	20	20
Tetradecane	17,245-6	31064	8.66	20	20
Hexadecane	29,631-7	31064	9.58	20	20
Octadecane	O-65-2	31064	10.5	20	20
Eicosane	21,927-4	31064	11.6	20	20
Docosane	13,445-7	31064	13.1	20	20
Hexacosane	24,168-7	31064	14.7	20	20
Tetracosane	T875-2	31064	16.3	20	20
Octacosane	O-50-4	31064	18.0	20	20
Dotriacontane	D22,310-7	n\a	22.5	Retention time	Retention time

TPH DRO			TOTAL =	200	200

(a) - conc. of standards may vary slightly based on actual weights from neat standards.

TABLE 3.
Initial Calibration Curve Concentrations

Diesel Calibration made from Restek XHc Diesel #2 Composite standard in of methylene chloride at 50,000 ug/mL. Serial dilutions are performed from this mix.

COMPOUNDS	STD 1 ug/mL	STD 2 ug/mL	STD 3 ug/mL	STD 4 ug/mL	STD 5 ug/mL	STD 6 ug/mL	STD 7 ug/mL
Diesel fuel #2	50,000	10,000	2,000	400	80	16	3.2

units are in ug/mL

TABLE 4..
Precision and Accuracy Limits for LCS
for Diesel Range Organics (DRO) Petroleum Hydrocarbons.

COMPOUNDS	Water Accuracy %R	Water Precision RPD		Soil Accuracy %R	Soil Precision RPD
TPH GC DRO	71.3-116	0-15		66.8-113	0-33

TABLE 5.
Precision and Accuracy Limits for MS and MSD
for Diesel Range Organics (DRO) Petroleum Hydrocarbons.

COMPOUNDS	Water Accuracy %R	Water Precision RPD		Soil Accuracy %R	Soil Precision RPD
TPH GC DRO	48-121	0-15		41.9-137	0-33

TABLE 6.
Method Detection Limits and Reporting Limits
for Diesel Range Organics (DRO) Petroleum Hydrocarbons.

COMPOUNDS	Water MDL (mg/L)	Water MRL (mg/L)	Soil MDL (mg/Kg)	Soil MRL (mg/Kg)
TPH GC DRO	0.38	1.0	0.92	4.0

Attachment QAPP-B5

Semivolatile Organic Compounds by GC/MS
Method 8270C

GAM 8270B

Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique

Revision 6.1 : 02/05/02

1.0 SCOPE AND APPLICATION

1.1 GAM8270B is used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. Direct injection of a sample may be used in limited applications. The following compounds can be determined by this method:

ANALYTE:	3510	3520	3540	3550	3580	CAS #
Acenaphthene	X	X	X	X	X	83-32-9
Acenaphthene-d(10) (I.S.)	X	X	X	X	X	
Acenaphthylene	X	X	X	X	X	208-96-8
Anthracene	X	X	X	X	X	120-12-7
Benzidine	CP	CP	CP	CP	CP	92-87-5
Benzoic acid	X	X	ND	X	X	65-85-0
Benz(a)anthracene	X	X	X	X	X	56-55-3
Benzo(b)fluoranthene	X	X	X	X	X	205-99-2
Benzo(k)fluoranthene	X	X	X	X	X	207-08-9
Benzo(g,h,i)perylene	X	X	X	X	X	191-24-2
Benzo(a)pyrene	X	X	X	X	X	50-32-8
Benzyl alcohol	X	X	ND	X	X	100-51-6
Bis(2-chloroethoxy)methane	X	X	X	X	X	111-91-1
Bis(2-chloroethyl)ether	X	X	X	X	X	111-44-4
Bis(2-chloroisopropyl)ether	X	X	X	X	X	39638-32-9
Bis(2-ethylhexyl)phthalate	X	X	X	X	X	117-81-7
4-Bromophenyl phenyl ether	X	X	X	X	X	101-55-3
Butyl benzyl phthalate	X	X	X	X	X	85-68-7
4-Chloroaniline	X	ND	ND	ND	X	106-47-8
4-Chloro-3-methylphenol	X	X	X	X	X	59-50-7
2-Chloronaphthalene	X	X	X	X	X	91-58-7
2-Chlorophenol	X	X	X	X	X	95-57-8
4-Chlorophenyl phenyl ether	X	X	X	X	X	7005-72-3
Chrysene	X	X	X	X	X	218-01-9
Chrysene-d(12) (I.S.)	X	X	X	X	X	
Dibenz(a,h)anthracene	X	X	X	X	X	53-70-3
Dibenzofuran	X	X	ND	X	X	132-64-9
Di-n-butylphthalate	X	X	X	X	X	84-74-2
1,2-Dichlorobenzene	X	X	X	X	X	95-50-1
1,3-Dichlorobenzene	X	X	X	X	X	541-73-1
1,4-Dichlorobenzene	X	X	X	X	X	106-46-7
1,4-Dichlorobenzene-d(4)(I.S)	X	X	X	X	X	
3,3'-Dichlorobenzidine	X	X	X	X	X	91-94-1
2,4-Dichlorophenol	X	X	X	X	X	120-83-2
2,6-Dichlorophenol	X	ND	ND	ND	X	87-65-0
Diethyl phthalate	X	X	X	X	X	84-66-2
2,4-Dimethylphenol	X	X	X	X	X	105-67-9
Dimethyl phthalate	X	X	X	X	X	131-11-3
2,4-Dinitrophenol	X	X	X	X	X	51-28-5
2,4-Dinitrotoluene	X	X	X	X	X	121-14-2
2,6-Dinitrotoluene	X	X	X	X	X	606-20-2
Diphenylamine	X	X	X	X	X	122-39-4
Di-n-octylphthalate	X	X	X	X	X	117-84-0

Fluoranthene	X	X	X	X	X	206-44-0
Fluorene	X	X	X	X	X	86-73-7
2-Fluorobiphenyl (surr.)	X	X	X	X	X	321-60-8

ANALYTE:	3510	3520	3540	3550	3580	CAS #
2-Fluorophenol (surr.)	X	X	X	X	X	367-12-4
Hexachlorobenzene	X	X	X	X	X	118-74-1
Hexachlorobutadiene	X	X	X	X	X	87-68-3
Hexachlorocyclopentadiene	X	X	X	X	X	77-47-4
Hexachloroethane	X	X	X	X	X	67-72-1
Indeno(1,2,3-cd)pyrene	X	X	X	X	X	193-39-5
Isophorone	X	X	X	X	X	78-59-1
2-Methylnaphthalene	X	X	ND	X	X	91-57-6
2-Methylphenol	X	ND	ND	ND	X	95-48-7
3-Methylphenol	X	ND	ND	ND	X	108-39-4
4-Methylphenol	X	ND	ND	ND	X	106-44-5
Naphthalene	X	X	X	X	X	91-20-3
Naphthalene-d(8) (I.S.)	X	X	X	X	X	
2-Nitroaniline	X	X	ND	X	X	88-74-4
3-Nitroaniline	X	X	ND	X	X	99-09-2
4-Nitroaniline	X	X	ND	X	X	100-01-6
Nitrobenzene	X	X	X	X	X	98-95-3
Nitrobenzene-d(5) (surr.)	X	X	X	X	X	
2-Nitrophenol	X	X	X	X	X	88-75-5
4-Nitrophenol	X	X	X	X	X	100-02-7
N-Nitrosodimethylamine	X	X	X	X	X	62-75-9
N-Nitrosodiphenylamine	X	X	X	X	X	86-30-6
N-Nitrosodi-n-propylamine	X	X	X	X	X	621-64-7
Pentachlorophenol	X	X	X	X	X	87-86-5
Perylene-d(12) (I.S.)	X	X	X	X	X	
Phenanthrene-d(10) (I.S.)	X	X	X	X	X	
Phenol	DC(28)	X	X	X	X	108-95-2
Phenol-d(6) (surr.)	DC(28)	X	X	X	X	
Pyrene	X	X	X	X	X	129-00-0
Pyridine	ND	ND	ND	ND	ND	110-86-1
Terphenyl-d(14)(surr.)	X	X	ND	X	X	
2,4,6-Tribromophenol(surr.)	X	X	X	X	X	
1,2,4-Trichlorobenzene	X	X	X	X	X	120-82-1
2,4,5-Trichlorophenol	X	X	ND	X	X	95-95-4
2,4,6-Trichlorophenol	X	X	X	X	X	88-06-2
Aldrin	X	X	X	X	X	309-00-2 *
4-Aminobiphenyl	X	ND	ND	ND	X	92-67-1 *
Aniline	X	X	ND	X	X	62-53-3 *
Aroclor - 1016	X	X	X	X	X	12674-11-2 *
Aroclor - 1221	X	X	X	X	X	11104-28-2 *
Aroclor - 1232	X	X	X	X	X	11141-16-5 *
Aroclor - 1242	X	X	X	X	X	346689-21-9 *
Aroclor - 1248	X	X	X	X	X	12672-29-6 *
Aroclor - 1254	X	X	X	X	X	11097-69-1 *
Aroclor - 1260	X	X	X	X	X	11096-82-5 *

alpha-BHC	X	X	X	X	X	319-84-6	*
beta-BHC	X	X	X	X	X	319-85-7	*
delta-BHC	X	X	X	X	X	319-86-8	*
gamma-BHC Lindane	X	X	X	X	X	58-89-9	*
Chlordane	X	X	X	X	X	57-74-9	*
4,4'-DDD	X	X	X	X	X	72-54-8	*
4,4'-DDE	X	X	X	X	X	72-55-9	*
4,4'-DDT	X	X	X	X	X	50-29-3	*
Dibenzo(a,e)pyrene	ND	ND	ND	ND	X	192-65-4	*
Dieldrin	X	X	X	X	X	60-57-1	*
1,2-Diphenylhydrazine	X	X	X	X	X	122-66-7	*

ANALYTE:	3510	3520	3540	3550	3580	CAS #	
Endosulfan I	X	X	X	X	X	959-98-8	*
Endosulfan II	X	X	X	X	X	33212-65-9	*
Endosulfan sulfate	X	X	X	X	X	1031-07-8	*
Endrin	X	X	X	X	X	72-20-8	*
Endrin aldehyde	X	X	X	X	X	7421-93-4	*
Endrin ketone	X	X	ND	X	X		*
Heptachlor	X	X	X	X	X	76-44-8	*
Heptachlor epoxide	X	X	X	X	X	1024-57-3	*
Methoxychlor	X	ND	ND	ND	X	72-43-5	*
3-Methylcholanthrene	X	ND	ND	ND	X	56-49-5	*
4,4'-Methylenebis(2-chloraniline)	OE,OS(0)	ND	ND	ND	L	101-14-4	*
4,4'-Methylenebis (N,N-dimethylaniline)	X	X	ND	ND	ND	101-61-1	*
1,4-Naphthoquinone	X	ND	ND	ND	X	130-15-4	*
N-Nitrosodibutylamine	X	ND	ND	ND	X	924-16-3	*
N-Nitrosodiethylamine	X	ND	ND	ND	X	55-18-5	*
N-Nitrosomethylethylamine	X	ND	ND	ND	X	10595-95-6	*
Toxaphene	X	X	X	X	X	8001-35-2	*

* = compounds may be run for confirmation, not routine 8270B.
(a) Chemical Abstract Service Registry Number
(AW) = Adsorption to walls of glassware during extraction and storage.
(CP) = Nonreproducible chromatographic performance.
(DC) = Unfavorable distribution coefficient (number in parenthesis is percent recovery).
(HE) = Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery).
(HS) = Hydrolysis-during storage (number in parenthesis is percent stability).
(LR) = Low response.
(ND) = Not determined.
(OE) = Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery).
(OS) = Oxidation during storage (number in parenthesis is percent

stability).

(X) = Greater than 70 percent recovery by this technique.

Percent Stability = Average Recovery (Day 7) x 100/Average Recovery (Day 0).

INSTRUMENTATION: GC/MS

1.2 GAM8270B can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for a list of compounds and their characteristic ions that have been evaluated on the specified GC/MS system.

1.3 The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor. Under the alkaline conditions of the extraction step, a-BHC, g-BHC, endosulfan I and II, and endrin are subject to decomposition. Neutral extraction should be performed if these compounds are expected. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol,4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.4 The method reporting limit (MRL) of Method 8270B for determining an individual compound is approximately 0.20 -1.00 mg/Kg(wet weight) for soil/sediment samples, 1-200 mg/Kg for wastes (dependent on matrix and method of preparation), and 5.0 ug/L for ground water samples (see Table 10). MRL's will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation and cleanup methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract and for their qualitative and quantitative analysis by mass spectrometry.

3.0 INTERFERENCES

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-concentration and low concentration samples are sequentially analyzed. To eliminate carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph/mass spectrometer system

4.1.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be

directly coupled to the source. Varian 3400 with 1077 (or 1078) spilt/splitless injector, or 1093 SPI (Septum Programmable Injector) injector.

4.1.2 Column - 30 m * 0.25 mm ID (or 0.32 mm ID) 0.25 um film thickness silicone-coated fused-silica capillary column (J&W Scientific DB-5MS or equivalent.)

4.1.3 Mass spectrometer - Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be tuned with decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 3 when 1-2 uL of the GC/MS tuning standard is injected through the GC (5 ng/uL of DFTPP).

4.1.4 GC/MS interface - Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

4.1.5 Data system - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file

for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available. SATURN II GC/MS software version 5.2 is currently being used to integrate and report data.

4.1.6 Guard column (optional) (4-6m of Alltech econo-cap EC-5 0.32mm x 0.25µ cat.#19646 - or equivalent) between the injection port and analytical column joined with an appropriate column connector.

4.2 Autosampler syringe - 10 uL.
4.3 Volumetric flasks, Class A - 10 mL to 1000 mL.
4.4 Balance - Analytical, 0.0001 g.
4.5 Autosampler vials - 1.8 mL glass with Teflon-lined screw caps.
4.6 Screw cap vials - 8 mL borosilicate glass.
4.7 Syringe - 100 uL Hamilton gas tight or equivalent.
4.8 Syringe - 1000 uL Unimetrics gas tight or equivalent.
4.9 Syringe - 10 uL Hamilton or equivalent.
4.10 Repipet - 1-5 mL.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

5.3.1 Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source. Or, Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard.

5.3.2 Transfer the stock standard solutions into bottles with Teflon lined screw-caps. Store at <-10°C and protect from light. Stock standard solutions should be checked frequently for signs of

degradation or evaporation, especially just prior to preparing calibration standards from them.

5.3.3 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

5.4 Internal standard solutions - The internal standards recommended are 1,4-dichlorobenzene-d(4), naphthalene-d(8), acenaphthene-d(10), phenanthrene-d(10), pyrene-d(10), and perylene-d(12) (see Table 5). Other compounds may be used as internal standards as long as the requirements given in Section 7.3.2 are met. Internal Standards solutions can also be purchased from certified sources (e.g. - RESTEK cat. #31206 2,000 ug/mL in MeCl2), or prepared from certified neat standards to a similar concentration. The resulting solution will contain each standard at a concentration of 2,000 ng/uL. Each 1 mL sample extract undergoing analysis should be spiked with 10 uL of the internal standard solution, resulting in a concentration of 20 ng/uL of each internal standard. Store at <-10°C when not being used.

5.5 GC/MS tuning standard - A methylene chloride solution containing 5 ng/uL of decafluorotriphenylphosphine (DFTPP) should be prepared. This solution can be prepared from a commercially available standards (e.g. - 2uL of RESTEK cat.# 31001 at 2,500 ug/mL in 1 mL MeCl2). The standard may also contain 50 ng/uL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance. Store at <-10°C when not being used.

5.6 Calibration standards - A minimum of five calibration standards should be prepared. One of the calibration standards should be at a concentration near, but above, the method detection limit; the others should correspond to the range of concentrations found in real samples but should not exceed the working range of the GC/MS system. (See Table 12). Each standard should contain each analyte for detection by this method (e.g. some or all of the compounds listed in Table 1 may be included). Each 1 mL aliquot of calibration standard should be spiked with 10 uL of the internal standard solution prior to analysis. All standards should be stored at -10°C to -20°C and should be freshly prepared once a year, or sooner if check standards indicate a problem.

5.7 Surrogate standards - The recommended surrogate standards are phenol-d(6), 2-fluorophenol, 2,4,6-tribromophenol (RESTEK #31087), nitrobenzene-d(5), 2-fluorobiphenyl, and p-terphenyl-d(14) (RESTEK #31086). See Method GAM3510 or GAM3550 for the instructions on preparing the surrogate standards. Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards in all blanks, spikes, and sample extracts. Take into account all dilutions of sample.

5.8 Matrix spike standards - See Method GAM3510 or GAM3550 for instructions on preparing the matrix spike standard. Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Inject this concentration into the GC/MS to determine recovery of surrogate standards in all matrix spikes. Take into account all dilutions of sample extracts.

5.9 Acetone, hexane, methylene chloride, isooctane, carbon disulfide, toluene, and other appropriate solvents - pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Refer to the SW846 Field Manual and SW846 Chapter 1.

7.0 PROCEDURE

7.1 Sample preparation - Samples must be prepared by one of the following methods prior to GC/MS analysis.

Matrix	Methods
Water	GAM 3510B
Soil/sediment	GAM 3550A, GAM 3545
Waste	GAM 3550A, GAM 3580, GAM 3545

7.1.1 Direct injection - In very limited applications direct injection of the sample into the GC/MS system with a 10 uL syringe may be appropriate. The detection limit is very high (approximately 10,000 ug/L); therefore, it is only permitted where concentrations in excess of 10,000 ug/L are expected. The system must be calibrated by direct injection.

7.2 Extract cleanup - Extracts may be cleaned up by any of the following methods prior to GC/MS analysis. GEO Analytical, Inc. can currently perform the following cleanup procedures:

Compounds	Methods
Phenols, Polynuclear aromatic hydrocarbons	GAM 3630B
Phthalate esters, Nitrosamines, Haloethers	GAM 3620A
Organochlorine pesticides & PCBs, Chlorinated hydrocarbons	GAM 3620A
Nitroaromatics, cyclic ketones, Organophosphorus pesticides	GAM 3620A
All priority pollutant base, neutral, and acids	n/a

7.3 Initial calibration - The recommended GC/MS operating conditions:

Mass range:	35-500 amu
Scan time:	1 sec/scan
Initial temperature:	35°C, hold for 1 minutes
Temperature program:	35°C-300°C at 10°C/min

Final temperature:	300°C, hold 10 min.
Injector temperature:	250°-315°C at 200°C/min. splitless.
Transfer line temperature:	310°C

Source temperature:	250°C
Injector:	1093 (SPI)
Sample volume:	1-2 uL
Valve time:	1.00 minutes
Carrier gas:	Helium at ~ 1mL/min.

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 3 for a 5 ng/uL injection of DFTPP. Analyses should not begin until all these criteria are met. Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. When analyzing for pesticides, degradation of DDT to DDE and DDD should not exceed 20%. If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 in. of the capillary column.

7.3.2 The internal standards selected in Section 5.4 should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d(4) use m/z 152 for quantitation).

7.3.3 Analyze 1-2 uL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1). Calculate response factors (RFs) for each compound as follows:

$$RF = (A(x)C(is))/(A(is)C(x))$$

where:

A(x)= Area of the characteristic ion for the compound being measured.
A(is)= Area of the characteristic ion for the specific internal standard.
C(is) = Concentration of the specific internal standard (ng/uL).
C(x) = Concentration of the compound being measured (ng/uL).

7.3.4 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitro-phenol; and 4-nitrophenol. The minimum acceptable average

RF for these compounds is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. These compounds are continually monitored in each CCC for minimum requirements.

7.3.4.1 The percent relative standard deviation (%RSD=100[SD/RF]) should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) (see Table 4) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.4.2 If the %RSD of any CCC is 30% or greater, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with section 7.4.

7.3.5 Linearity - If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation (Section 7.6.2).

7.3.5.1 If the %RSD of any compound is greater than 15%, construct calibration curves of area ratio (A/Ais) versus concentration using first or second order regression fit of the five calibration points. The analyst should select the regression order which introduces the least calibration error into the quantitation (Section 7.6.2.2 and 7.6.2.3). If the %RSD is <15%, use of calibration curves is a recommended alternative to average response factor calibration, and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.

7.4 Daily GC/MS calibration

7.4.1 Prior to analysis of samples, the GC/MS tuning standard must be analyzed. A 5 ng/uL injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 3. These criteria must be demonstrated during each 12 hour shift.

7.4.2 A calibration standard(s) at 10 ng/uL, containing all semivolatile analytes, including all required surrogates, must be analyzed every 12 hours during analysis. Compare the instrument response

factor from the standards every 12 hours with the SPCC (Section 7.4.3) and CCC (Section 7.4.4) criteria.

7.4.3 System Performance Check Compounds (SPCCs): A system performance check must be made during every 12 hour shift. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

7.4.4 Calibration Check Compounds (CCCs): After the System performance check is met, all analytes are used to check the validity of the initial calibration.

Calculate the percent drift using:

$$\% \text{ Drift} = \frac{C(I) - C(c)}{C(I)} \times 100$$

where:

C(I) = Calibration Check Compound standard concentration.
C(c) = Measured concentration using selected quantitation method.

If the percent difference for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% drift) for any one compound, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated. This criterion must be met before sample analysis begins.

7.4.5 The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two for any sample, blank or standard (-50% to +100%) from the last daily calibration standard check, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. All samples analyzed during the malfunction must be re-analyzed.

7.5 GC/MS analysis

7.5.1 It is highly recommended that the extract be screened on a GC/FID or GC/PID using the same type of capillary column. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

7.5.2 Spike the 1 mL extract obtained from sample preparation with 10 uL of the internal standard solution just prior to analysis.

7.5.3 Analyze the 1 mL extract by GC/MS using a 30 m * 0.25 mm (or 0.32 mm) silicone-coated fused-silica capillary column. The volume to be injected should ideally contain 10 ng of base/neutral and 15 ng of acid surrogates (for a 1 uL injection). The recommended GC/MS operating conditions to be used are specified in Section 7.3. However, these conditions may be optimized on an ongoing basis.

7.5.4 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 20 ng/uL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

7.5.5 Perform all qualitative and quantitative measurements as described in Section 7.6. Store the extracts at 4-C, protected from light in screw-cap vials equipped with unpierced Teflon lined septa.

7.5.6 Record the sample numbers and order run in the sequence logbook for each run sequence. The resulting peak sizes in area units are saved by the data system software and processed using an automated integration/calibration package.

7.6 Data interpretation

7.6.1 Qualitative analysis

7.6.1.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

7.6.1.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.1.2 The RRT of the sample component is within +/- 0.06 RRT units of the RRT of the standard component.

7.6.1.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria can be met,

but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste delisting requirements may

require the reporting of nontarget analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
(2) The relative intensities of the major ions should agree within +/- 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.6.2 Quantitative analysis

7.6.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion.

7.6.2.2 If the %RSD of a compounds relative response factor is 15% or less, then the concentration in the extract may be determined using the average response factor average of (RF) from initial calibration data (7.3.5.) and the following equation:

$$C_{ex}\ (mg/L) = (A_x \times C_{is})/(A_{is} \times \overline{RF})$$

where: C_{ex} is the concentration of the compound in the extract (without considering the volume or weight), and the other terms are as defined in Section 7.3.3

7.6.2.3 Alternatively, the regression line fitted to the initial calibration (Section 7.3.5.1) may be used for determination of the extract concentration.

7.6.2.4 Compute the concentration of the analyte in the sample using the equations in Sections 7.6.2.4.1 and 7.6.2.4.2.

7.6.2.4.1 The concentration of the analyte in the liquid phase of the sample is calculated using the concentration of the

analyte in the extract and the volume of liquid extracted, as follows:

$$\text{Concentration in liquid (ug/L)} = \frac{(C_{ex} \times V_{ex})}{V_o} \times D$$

where:

C_{ex} = extract volume, in mL
V_{ex} = volume of liquid extracted, in L.
D = dilution factor

7.6.2.4.2 The concentration of the analyte in the solid phase of the sample is calculated using the concentration of the pollutant in the extract and the weight of the solids, as follows:

$$\text{Concentration as solid (ug/kg)} = \frac{(C_{ex} \times V_{ex})}{W_s \times D_s} \times D$$

where:

V_{ex} = extract volume, in mL
W_s = sample weight, in kg.
D = dilution factor
D_s = 1 for wet weight, or S/100 where :

$$S = \% \text{ solids} = \frac{\text{dry weight of sample}}{\text{wet weight of sample}} \times 100$$

7.6.2.5 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulas given above should be used with the following modifications: The areas A(x) and A(is) should be from the total ion chromatograms and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

7.6.2.6 Quantitation of multicomponent compounds (e.g. Aroclors) is beyond the scope of Method 8270B. Normally, quantitation is performed using a GC/ECD by Method 8080 or 8081.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent.

8.1.1 Calculate the values for the preparation blanks, laboratory control samples (LCS), matrix spikes(MS) and matrix spike duplicates(MSD)

8.1.1.1 If the preparation blank shows no contamination above the reporting limits for the analytes of interest, the method is presumed in control and sample analysis can proceed.

8.1.1.2 If the preparation blank contains contamination above the reporting limit, corrective actions must be performed to bring the method back into control. After the corrective actions are performed

the analyst(s) must demonstrate that the preparation and analysis procedures are free of contaminants before sample analysis can proceed.

8.1.1.3 Calculate the spike recoveries for the LCS, MS and MSD. If all recoveries are within the established limits in tables 8 & 9. The method is presumed in control and sample analysis can proceed.

8.1.1.4 If the spike recoveries for the LCS are within the established control limits in table 8 & 9, but the MS (and/or MSD)are not within the established limits in table 8 & 9, the method is presumed in control and sample analysis can proceed. Sample data for the spiked sample with recoveries outside of the acceptance limits in table 8 & 9 should be flagged as "estimated concentration."

8.1.1.5 If the spike recoveries for the LCS are not within the established control limits in table 8 & 9, corrective actions must be performed to bring the method back into control. After corrective actions are performed, the analyst(s) must demonstrate LCS recoveries within the established limits before sample analysis can proceed.

8.2 Gas Chromatographic System Check

8.2.1 The quality control check sample concentrate should contain each analyte at a concentration of 10 ug/mL in methylene chloride.

8.2.2 Table 6 indicates the calibration and QC acceptance criteria for method 8270B. Table 7 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables should be used to evaluate the laboratory's ability to perform and generate acceptable data by this method.

8.3 Retention time windows(also refer to GQAP 003)

8.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures and multi-response products (i.e. PCBs) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

8.3.2 Calculate the standard deviation of the three absolute retention times (use any function of retention time; including absolute retention time, or relative retention time) for each single component standard. For multi-response products, choose one major peak from the envelope and calculate the standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in samples.

8.3.2.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst

should weigh heavily in the interpretation of chromatograms. For multi-response analytes (i.e. PCBs), the

analyst should use the retention time window, but should primarily rely on pattern recognition.

8.3.2.2 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

8.3.3 The analyst must calculate retention time windows for each analyte on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

8.4 *Corrective Actions*

8.4.1 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within laboratory established limits in table 8. If a surrogate recovery is not within limits, the following procedures are required.

8.4.1.1 Check to be sure there are no errors in calculations, surrogate solutions. Also, check instrument performance. You may use the criteria in section 8.2 above.

8.4.1.2 Recalculate the data and/or re-analyze the extract if any of the above checks reveal a problem.

8.4.1.3 Re-extract and re-analyze the sample, if none of the above are a problem or flag the data as "estimated concentration."

8.4.1.4 Laboratory control samples and preparation blanks must have surrogate recoveries within laboratory established limits.

8.4.2 If the laboratory control sample (LCS) is out of control the following procedures are required.

8.4.2.1 Check to be sure there are no errors in calculations, spiking solutions. Also, check instrument performance.

8.4.2.2 Recalculate the data and/or re analyze the extract if any of the above checks reveal a problem.

8.4.2.3 Re-analyze the LCS sample to demonstrate that the analysis is in control.

8.4.2.4 Re-extract and re-analyze the LCS and all samples associated with the unacceptable LCS.

8.4.3 Samples that are prepared and run with an out of control preparation blank must be re-extracted and re-run along with a new preparation blank.

8.4.4 Flag data from samples that have unacceptable matrix spike recoveries as estimated concentration.

9.0 METHOD PERFORMANCE

9.1 Each laboratory that uses these methods is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control reference sample must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

9.2 Before processing any samples, the analyst should demonstrate, through the analysis of a reagent blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent blank should be processed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

9.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any major changes are made to the system (e.g. column changed), recalibration of the system must take place.

9.4 Required instrument QC is found in the following sections

9.4.1 The GC/MS system must be tuned to meet the DFTPP specification in Steps 7.3.1 and 7.4.1.

9.4.2 There must be an initial calibration of the GC/MS system as specified in step 7.3.

9.4.3 The GC/MS system must meet the SPCC criteria specified in Section 7.4.3 and the CCC criteria in Section 7.4.4, each 12 hours.

9.5 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

9.5.1 A quality control (QC) reference sample concentrate is required containing each analyte at a concentration of 100 mg/L in acetone. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC reference sample concentrate must be made using stock standards prepared independently from those used for calibration.

9.5.2 Using a pipet, prepare QC reference samples at a concentration of 100 ug/L by adding 1.00 mL of QC reference sample concentrate to each of four 1-L aliquots water.

9.5.3 Analyze the well-mixed QC reference samples according to the method beginning in Section 7.1 with extraction of the samples.

9.5.4 Calculate the average recovery of (x) in ug/L, and the standard deviation of the recovery (s) in ug/L, for each analyte of interest using the four results.

9.5.5 For each analyte, compare s and average of (x) with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and average of (x) for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual average of (x) falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

NOTE: The large number of analytes in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method is analyzed.

9.5.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 9.5.6.1 or 9.5.6.2.

9.5.6.1 Locate and correct the source of the problem and repeat the test for all analytes of interest beginning with Step 9.5.2.

9.5.6.2 Beginning with Step 9.5.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Step 9.5.2.

9.6 The laboratory must, on an ongoing basis, analyze a reagent blank, a Laboratory Control Sample (LCS), a matrix spike, and a matrix spike replicate for each analytical batch (up to maximum of 20 samples/batch) to assess accuracy. For soil and waste samples where detectable amounts of organics are present, replicate samples may be appropriate in place of matrix spiked samples. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

9.6.1 The concentration of the spike in the sample should be determined as follows:

9.6.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Step 9.6.2, whichever concentration would be larger.

9.6.1.2 If the concentration of a specific analyte in a water sample is not being checked against a limit specific to that analyte, the spike should be at 100 ug/L or 1 to 5 times higher than the background concentration determined in Step 9.6.2, whichever concentration would be larger. For other matrices, recommended spiking concentration is 20 times the EQL.

9.6.1.3 If it is impractical to determine background levels before spiking (e.g. maximum holding times will be exceeded), the spike concentration should be at (1) the regulatory concentration limit, if any; or, if none (2) the larger of

either 5 times higher than the expected background concentration or 100 ug/L. For other matrices, recommended spiking concentration is 20 times the EQL.

9.6.2 Analyze one sample aliquot to determine the background concentration (B) of each analyte. If necessary, prepare a new QC reference sample concentrate (Step 9.5.1) appropriate for the background concentration in the sample. Spike a second sample aliquot with 1.00 mL of the QC reference sample concentrate and analyze it to determine the concentration after spiking (A) of each analyte. Calculate each percent recovery (p) as 100(A- B)%T, where T is the known true value of the spike.

9.6.3 Compare the percent recovery (p) for each analyte in the water sample with the corresponding QC acceptance criteria found in Table 9. These acceptance criteria were calculated to include an allowance for error in the measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1. If spiking was performed at a concentration lower than 100ug/L, the analyst must use either the QC acceptance criteria presented in Table 6, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of an analyte: (1) Calculate accuracy (x') using the equation found in Table 7, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 7, substituting x' for average of(x); (3) calculate the range for recovery at the spike concentration as (100x'/T) +/- 2.44(100S'/T%.

9.6.4 If any individual p falls outside the designated range for recovery, that analyte has failed the acceptance criteria. A check standard containing each analyte that failed the criteria must be analyzed as described in Step 9.7

9.7 If any analyte in a water sample fails the acceptance criteria for recovery in Step 9.6, a QC reference sample containing each analyte that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC reference sample will depend upon the number of analytes being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of analytes in Table 6 must be measured in the sample in Step 9.6, the probability that the analysis of a QC reference sample will be required is high. In this case the QC reference sample should be routinely analyzed with the spiked sample.

9.7.1 Prepare the QC reference sample by adding 1.0 mL of the QC reference sample concentrate (Step 9.5.1 or 9.6.2) to 1 L of

water. The QC reference sample needs only to contain the analytes that failed criteria in the test in Step 9.6.

9.7.2 Analyze the QC reference sample to determine the concentration measured (A) of each analyte. Calculate each percent recovery (p(s)) as 100(A/T)%, where T is the true value of the standard concentration.

9.7.3 Compare the percent recovery (p(s)) for each analyte with the corresponding QC acceptance criteria found in Table 6. Only analytes that failed the test in Step 9.6 need to be compared with these criteria. If the recovery of any such analyte falls outside the designated range, the laboratory performance for that analyte is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that analyte in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

9.8 As part of the QC program for the laboratory, method accuracy for each matrix studied must be assessed and records must be maintained. After the analysis of five spiked samples (of the same matrix) as in Step 9.6, calculate the average percent recovery average of (p) and the standard deviation of the percent recovery (s(p)). Express the accuracy assessment as a percent recovery interval from average of (p) - 2s(p) to average of (p) + 2s(p). If average of (p) = 90% and s(p) = 10%, for example, the accuracy interval is expressed as 70-110%. Update the accuracy assessment for each analyte on a regular basis (e.g. after each five to ten new accuracy measurements).

9.9 To determine acceptable accuracy and precision limits for surrogate standards the following procedure should be performed.

9.9.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

9.9.2 Once a minimum of thirty samples of the same matrix have been analyzed, calculate the average percent recovery (P) and standard deviation of the percent recovery (s) for each of the surrogates.

9.9.3 For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard. This should be done as follows:

Upper Control Limit (UCL) = p + 3S
Lower Control Limit (LCL) = p - 3s

9.9.4 For aqueous and soil matrices, these laboratory established surrogate control limits should, if applicable, be compared with the control limits listed in Table 8. The limits given in Table 8 are GEO Analytical's limits for soil and aqueous samples, and therefore, the single-laboratory limits established in Step 9.9.3 must fall within those given in Table 8 for these matrices.

9.9.5 If recovery is not within limits, the following procedures are required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Re-extract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration".

9.9.6 At a minimum, each laboratory should update surrogate recovery limits on a matrix-by-matrix basis, annually.

9.10 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or a mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

10.0 REFERENCES

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9. Lopez-Avila, V. (W. Beckert, Project Officer); "Development of a Soxtec Extraction Procedure for Extraction of Organic Compounds from Soils and Sediments"; U.S. Environmental Protection Agency. Environmental Monitoring and Support Laboratory. Las Vegas, NV,October 1991; EPA 600/X-91/140.

TABLE 1a: Characteristic Ions For Semivolatile Compounds (Reported)

Compound	*Retention Time (min)	Primary Quan Ion	Secondary Quan Ion(s)
Phenol	6.33	94	65,66
Bis(2-chloroethyl) ether	6.41	63	94,95
2-Chlorophenol	6.51	128	64,130
1,3-Dichlorobenzene	6.81	146	148,111
1,4-Dichlorobenzene-d(4)I.S.)	6.99	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	7.39	107+108	79,77
1,2-Dichlorobenzene	7.36	146	148,111
Bis(2-chloroisopropyl) ether	7.68	45	77,121
N-Nitrosodi-n-propylamine	8.05	70	42,101,130
Hexachloroethane	8.18	117	201,199
Nitrobenzene	8.44	77	123,65
Isophorone	9.10	82	95,138
2-Nitrophenol	9.31	109	109,65,139
2,4-Dimethylphenol	9.59	107	107,121,122
Bis(2-chloroethoxy)methane	9.79	63	95,123
Benzoic acid	10.03	105	122,77
2,4-Dichlorophenol	10.04	162	164,98
1,2,4-Trichlorobenzene	10.19	180	182,145
Naphthalene-d(8)I.S.)	10.33	136	68
Naphthalene	10.40	128	129,127
Hexachlorobutadiene	10.78	225	223,227
4-Chloro-3-methylphenol	12.33	107	144,142
2-Methylnaphthalene	12.46	141	142
2-Methylphenol	7.74	107+108	107,108,77,79,90
Hexachlorocyclopentadiene	12.94	237	235,272
4-Methylphenol	8.18	107+108	107,108,77,79,90
2,4,6-Trichlorophenol	13.46	196	198,200
3-Methylphenol	8.18	107+108	107,108,77,79,90
2-Chloronaphthalene	13.95	162	127,164
2-Nitroaniline	14.44	138	92,138,65
Dimethyl phthalate	15.04	163	194,164
Acenaphthylene	15.25	152	151,153
2,6-Dinitrotoluene	15.24	165	63,89
3-Nitroaniline	15.76	65	108,92
Acenaphthene-d(10)I.S.)	15.68	162	164,160
Acenaphthene	15.78	153	154,152
2,4-Dinitrophenol	16.11	184	63,154
4-Chloroaniline	10.68	127	127,129,65,92
Dibenzofuran	16.33	168	139
2,4-Dinitrotoluene	16.51	165	63,89
4-Nitrophenol	16.61	139	109,65
Diethyl phthalate	17.28	149	177,150
Fluorene	17.41	166	165,167
4-Chlorophenyl phenyl ether	17.49	141	206,141,204
4,6-Dinitro-2-methylphenol	17.78	198	51,105
Diphenylamine	17.93(a)	169	168,167
4-Bromophenyl phenyl ether	19.01	141	250,248,141
2,4,5-Trichlorophenol	13.59	196	196,198,97,132,99
Hexachlorobenzene	19.13	284	142,249
Pentachlorophenol	19.86	266	264,268

4-Nitroaniline	17.69	138	138,65,108,92, 80,39
Phenanthrene-d(10)(i.s.)	20.34	188	94,80
Phenanthrene	20.48	178	179,176
Anthracene	20.58	178	176,179
Carbazole	21.23	167	166,139
Di-n-butylphthalate	22.43	149	150,104

Fluoranthene	24.18	202	101,203
Pyrene	24.88	202	200,203
Pyrene-d10 (I.S)	24.81	212	211,213
Butyl benzyl phthalate	27.14	149	91,206
Benz(a)anthracene	28.63	228	229,226
3,3'-Dichlorobenzidine	28.69	252	254,126
Chrysene	28.73	228	226,229
Bis(2-ethylhexyl)phthalate	28.88	149	167,279
Di-n-octyl phthalate	30.53	149	167,43
Benzo(b)fluoranthene	31.34	252	253,125
Benzo(k)fluoranthene	31.41	252	253,125
Benzo(a)pyrene	32.09	252	253,125
Perylene-d(12)I.S.)	32.24	264	260,265
Indeno(1,2,3-cd)pyrene	35.08	276	138,227
Dibenz(a,h)anthracene	35.13	278	139,279
Benzo[g,h,i]perylene	35.91	276	138,277
2-Fluorobiphenyl (surr.)	13.66	172	171
2-Fluorophenol (surr.)	4.43	112	64
Nitrobenzene-d(5)surr.)	8.39	82	128,54
N-Nitrosodimethylamine	2.71	73+74+75	44
Phenol-d(6) (surr.)	6.29	99	42,71
Terphenyl-d(14) (surr.)	25.50	244	122,212
2,4,6-Tribromophenol (surr.)	18.21	332	330,141

I.S. = internal standard.
surr. = surrogate.
(a) Estimated retention times.
(b) Substitute for the non-specific mixture, tricresyl phosphate.
* Retention times may vary depending on column or instrument used.

TABLE 1b: Characteristic Ions For Semivolatile Compounds (Possible or for Conformation)

Compound	*Retention Time (min)	Primary Quan Ion	Secondary Quan Ion(s)
Aldrin	--	66	263,220
Aroclor-1016	--	222	260,292
Aroclor-1221	--	190	224,260
Aroclor-1232	--	190	224,260
Aroclor-1242	--	222	256,
Aroclor-1248	--	292	362,32
Aroclor-1254	--	292	362,326
alpha-BHC	--	183	181,109
beta-BHC	--	181	183,109
delta-BHC	--	183	181,109
gamma-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Toxaphene	--	159	231,233
Carbofuran	24.90	164	164,149,131,122
N-Nitrosodibutylamine	16.73	84	84,57,41,116,158
Aniline	5.68	93	66,65
Pyridine	2.74	79+80	
7,12-Dimethylbenz(a)anthracene	33.25	256	256,241,239,120
Benzidine	23.87	184	92,185
Chrysene-d(12)I.S.)	27.88	240	120,236
N-Nitrosomethylethylamine	6.97	88	42,88,43,56
Methoxychlor	33.55	227	227,228,152,114,

			274,212
Dibenz(a,j)acridine	36.40	279	279,280,277,250
N-Nitrosodiethylamine	8.70	102	102,42,57,44,56
4-Aminobiphenyl	25.08	169	169,168,170,115
1-Chloronaphthalene	13.65(a)	162	127,164
2,6-Dinitrophenol	15.47	162	162,164,126,98,63

I.S. = internal standard.
surr. = surrogate.
(a) Estimated retention times.
(b) Substitute for the non-specific mixture, tricresyl phosphate.
* Retention times may vary depending on column or instrument used.

Table 2: Equations Used for Semi-Volatiles GC/MS Internal Standard Quantitation

Parameter	Equation	Units
relative response factor (RRF)	$RRF = \frac{(A_s)\ (C_{is})}{(A_{is})\ (C_s)}$ where : A_s = area of the characteristic ion of the analyte being measured A_{is} = area of the characteristic ion of the specific internal standard C_{is} = concentration of the specific internal standard in nanograms (ng). C_s = concentration of the analyte being measured in nanograms (ng).	

Water concentration	$$\frac{(A_s)\ (C_{is})}{(A_{is})\ (RRF)\ (V_s)} \times d$$ where : A_x = area of the characteristic ion of the analyte being measured A_{is} = area of the characteristic ion of the specific internal standard C_{is} = concentration of the specific internal standard in nanograms (ng). RRF= response factor d = dilution factor, dimensionless V_s = initial sample volume in mL	µg/L
Soil concentration (wet weight)	$$\frac{(A_x)\ (C_{is})}{(A_{is})\ (RRF)\ (W_s)\ (D_s)} \times d$$ where : A_x = area of the characteristic ion of the analyte being measured A_{is} = area of the characteristic ion of the specific internal standard C_{is} = concentration of the specific internal standard in nanograms (ng). RRF= response factor d = dilution factor, dimensionless W_s = initial sample weight in grams D_s = 1 for wet weight, or S/100 where : S = % solids = $\frac{\text{dry weight of sampl}}{\text{wet weight of samp}} \times 100$	µg/g

TABLE 3: DFTPP Key Ions And Ion Abundance Criteria(a)*

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

(a) See Reference 4.

* alternate tuning criteria may be used (e.g. CLP, Method 525, or manufacturer's instructions.) provided method performance is not adversely affected.

TABLE 4: Calibration Check Compounds

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

TABLE 5. Semivolatile Internal Standards With Corresponding Analytes Assigned For Quantitation

1,4-Dichlorobenzene-d(4)	Naphthalene-d(8)	Acenaphthene-d(10)
Aniline		Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl)ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	
1,4-Dichlorobenzene	2,6-Dichlorophenol	Dibenzofuran
1,2-Dichlorobenzene		Diethyl phthalate
		Dimethyl phthalate
2-Fluorophenol (surr.)	2,4-Dimethylphenol	2,4-Dinitrophenol
Hexachloroethane	Hexachlorobutadiene	2,4-Dinitrotoluene
	Isophorone	2,6-Dinitrotoluene
2-Methylphenol	2-Methylnaphthalene	Fluorene
4-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr.)
N-Nitrosodimethylamine	Nitrobenzene	
N-Nitroso-di-n-propylamine	Nitrobenzene-d(8)(surr.)	Hexachlorocyclo-pentadiene
Phenol	2-Nitrophenol	
Phenol-d(6)(surr.)		
	1,2,4-Trichlorobenzene	2-Nitroaniline
		3-Nitroaniline
		4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetra-chlorobenzene
		2,3,4,6-Tetra-chlorophenol
		2,4,6-Tribromo-phenol (surr.)
		2,4,6-Trichloro-phenol
		2,4,5-Trichloro-phenol

Phenanthrene-d(10)	Pyrene-d(10)	Perylene-d(12)
	Benzidine	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl)phthalate	Benzo(g,h,i)perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
2,4,6-Tribromophenol	Chrysene	Di-n-octyl phthalate
Carbazole	3,3'-Dichlorobenzidine	Indeno(1,2,3-cd)pyrene
	Fluoranthene	Dibenz(a,h)anthracene
Diphenylamine	Pyrene	
Hexachlorobenzene	Terphenyl-d,(14)(surr.)	
Pentachlorophenol		
Azobenzene		
Phenanthrene		
4,6-Dinitro-2-methylphenol		
N-Nitrosodiphenylamine		

TABLE 6: QC Acceptance Criteria(a)

Compound	Test conc. (ug/L)	Limit for s (ug/L)	Range for x (ug/L)	Range p, p(s) (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Anthracene	100	32.0	43.4-118.0	27.133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzylbutyl phthalate	100	23.4	D-139.9	D-152
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octylphthalate	100	31.4	18.6-131.8	4-146
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147

2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

TABLE 6 (continued): QC Acceptance Criteria(a)

Compound	Test conc. (ug/L)	Limit for s (ug/L)	Range for x (ug/L)	Range p, p(s) (%)
Aldrin *	100	39.0	7.2-152.2	D-166
beta-BHC *	100	31.5	41.5-130.6	24-149
delta-BHC *	100	21.6	D-100.0	D-110
4,4'-DDD *	100	31.0	D-134.5	D-145
4,4'-DDE *	100	32.0	19.2-119.7	4-136
4,4'-DDT *	100	61.6	D-170.6	D-203
Dieldrin *	100	30.7	44.3-119.3	29-136
Endosulfan sulfate *	100	16.7	D-103.5	D-107
Endrin aldehyde *	100	32.5	D-188.8	D-209
Heptachlor *	100	37.2	D-172.2	D-192
Heptachlor epoxide *	100	54.7	70.9-109.4	26.155
PCB-1260 *	100	54.2	19.3-121.0	D-164

(s) = Standard deviation of four recovery measurements, in ug/L.

(x) = Average recovery for four recovery measurements, in ug/L.

(p,p(s)) = Percent recovery measured.

(D) = Detected; result must be greater than zero.

(a) = Criteria from 40 CFR Part 136 for Method 625. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

(*) = Compounds run for confirmation purposes only.

TABLE 7: Method Accuracy And Precision As Functions Of Concentration(a)

Compound	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s(r)' (ug/L)	Overall precision, S'(ug/L)
Acenaphthene	0.96C+0.19	0.15x-0.12	0.21x-0.67
Acenaphthylene	0.89C+0.74	0.24x-1.06	0.26x-0.54
Anthracene	0.80C+0.68	0.21x-0.32	0.27x-0.64
Benz(a)anthracene	0.88C-0.60	0.15x+0.93	0.26x-0.21
Benzo(b)fluoranthene	0.93C-1.80	0.22x+0.43	0.29x+0.96
Benzo(k)fluoranthene	0.87C-1.56	0.19x+1.03	0.35x+0.40
Benzo(a)pyrene	0.90C-0.13	0.22x+0.48	0.32x+1.35
Benzo(ghi)perylene	0.98C-0.86	0.29x+2.40	0.51x-0.44
Benzyl butyl phthalate	0.66C-1.68	0.18x+0.94	0.53x+0.92
Bis(2-chloroethyl) ether	0.86C-1.54	0.35x-0.99	0.35x+0.10
Bis(2-chloroethoxy)methane	1.12C-5.04	0.16x+1.34	0.26x+2.01
Bis(2-chloroisopropyl)ether	1.03C-2.31	0.24x+0.28	0.25x+1.04
Bis(2-ethylhexyl) phthalate	0.84C-1.18	0.26x+0.73	0.36x+0.67
4-Bromophenyl phenyl ether	0.91C-1.34	0.13x+0.66	0.16x+0.66
2-Chloronaphthalene	0.89C+0.01	0.07x+0.52	0.13x+0.34
4-Chlorophenyl phenyl ether	0.91C+0.53	0.20x-0.94	0.30x-0.46
Chrysene	0.93C-1.00	0.28x+0.13	0.33x-0.09
Dibenzo(a,h)anthracene	0.88C+4.72	0.30x+8.51	0.59x+0.25
Di-n-butyl phthalate	0.59C+0.71	0.13x+1.16	0.39x+0.60
1,2-Dichlorobenzene	0.80C+0.28	0.20x+0.47	0.24x+0.39
1,3-Dichlorobenzene	0.86C-0.70	0.25x+0.68	0.41x+0.11
1,4-Dichlorobenzene	0.73C-1.47	0.24x+0.23	0.29x+0.36
3,3'-Dichlorobenzidine	1.23C-12.65	0.28x+7.33	0.47x+3.45
Diethyl phthalate	0.43C+1.00	0.28x+1.44	0.52x+0.22
Dimethyl phthalate	0.20C+1.03	0.54x+0.19	1.05x-0.92
2,4-Dinitrotoluene	0.92C-4.81	0.12x+1.06	0.21x+1.50
2,6-Dinitrotoluene	1.06C-3.60	0.14x+1.26	0.19x+0.35
Di-n-octyl phthalate	0.76C-0.79	0.21x+1.19	0.37x+1.19
Fluoranthene	0.81C+1.10	0.22x-0.73	0.28x-0.60
Fluorene	0.90C-0.00	0.12x+0.26	0.13x+0.61
Hexachlorobenzene	0.74C+0.66	0.18x-0.10	0.43x-0.52
Hexachlorobutadiene	0.71C-1.01	0.19x+0.92	0.26x+0.49
Hexachloroethane	0.73C-0.83	0.17x+0.67	0.17x+0.80
Indeno(1,2,3-cd)pyrene	0.78C-3.10	0.29x+1.46	0.50x-0.44
Isophorone	1.12C+1.41	0.27x+0.77	0.33x+0.26
Naphthalene	0.76C+1.58	0.21x-0.41	0.30x-0.68
Nitrobenzene	1.09C-3.05	0.19x+0.92	0.27x+0.21
N-Nitrosodi-n-propylamine	1.12C-6.22	0.27x+0.68	0.44x+0.47
Phenanthrene	0.87C+0.06	0.12x+0.57	0.15x+0.25
Pyrene	0.84C-0.16	0.16x+0.06	0.15x+0.31
1,2,4-Trichlorobenzene	0.94C-0.79	0.15x+0.85	0.21x+0.39
4-Chloro-3-methylphenol	0.84C+0.35	0.23x+0.75	0.29x+1.31
2-Chlorophenol	0.78C+0.29	0.18x+1.46	0.28x+0.97
2,4-Dichlorophenol	0.87C-0.13	0.15x+1.25	0.21x+1.28
2,4-Dimethylphenol	0.71C+4.41	0.16x+1.21	0.22x+1.31
2,4-Dinitrophenol	0.81C-18.04	0.38x+2.36	0.42x+26.29
2-Methyl-4,6-dinitrophenol	1.04C-28.04	0.10x+42.29	0.26x+23.10
2-Nitrophenol	0.07C-1.15	0.16x+1.94	0.27x+2.60
4-Nitrophenol	0.61C-1.22	0.38x+2.57	0.44x+3.24
Pentachlorophenol	0.93C+1.99	0.24x+3.03	0.30x+4.33

Phenol	0.43C+1.26	0.26x+0.73	0.35x+0.58
2,4,6-Trichlorophenol	0.91C-0.18	0.16x+2.22	0.22x+1.81
Aldrin *	0.78C+1.66	0.27x-1.28	0.43x+1.13
beta-BHC *	0.87C-0.94	0.20x-0.58	0.30x+1.94

TABLE 7 (continued): Method Accuracy And Precision As Functions Of Concentration(a)

Compound	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s(r)' (ug/L)	Overall precision, S'(ug/L)
delta-BHC *	0.29C-1.09	0.34x+0.86	0.93x-0.17
4,4'-DDD *	0.56C-0.40	0.29x-0.32	0.66x-0.96
4,4'-DDE *	0.70C-0.54	0.26x-1.17	0.39x-1.04
4,4'-DDT *	0.79C-3.28	0.42x+0.19	0.65x-0.58
Dieldrin *	0.82C-0.16	0.20x-0.16	0.26x-0.07
Endosulfan sulfate *	0.39C+0.41	0.12x+2.47	0.63x-1.03
Endrin aldehyde *	0.76C-3.86	0.18x+3.91	0.73x-0.62
Heptachlor *	0.87C-2.97	0.24x-0.56	0.50x-0.23
Heptachlor epoxide *	0.92C-1.87	0.33x-0.46	0.28x+0.64
PCB-1260 *	0.81C-10.86	0.35x+3.61	0.43x+1.82

(x)' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

(s(r)' = Expected single analyst standard deviation of measurements at an average concentration of x, in ug/L.

(S)' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in ug/L.

(C) = True value for the concentration, in ug/L.

(x) = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

(*) = Compounds run for confirmation.

TABLE 8: Surrogate Spike Recovery Limits For Water And Soil/Sediment Samples based on data generated by Geo Analytical, Inc.

METHOD 8270B Surrogates	Acceptable Range for Water%	Acceptable Range for Soil%
2-Fluorophenol	11-62.3	25-121
Phenol d6	2.9-44.4	24-113
2,4,6-Tribromophenol	28.4-114	19-122
2-Chlorophenol d4	33-110*	20-130*
1,2-Dichlorobenzene d4	16-110*	20-130*
Nitrobenzene d5	15.3-127	23-120
2-Fluorobiphenyl	16.5-115	30-115
Terphenyl d14	24.7-109	18-137

* not reported at this time

TABLE 9: LCS,MS & MSD Recovery Limits For Water And Soil/Sediment Samples

SEMI-VOLATILE ORGANICS BY GC/MS

COMPOUNDS	Water Method (EPA/SW846)	Water LCS/MS Recovery	Water RPD	Soil Method (SW846)	Soil LCS Recovery	Soil MS/MSD Recovery	Soil RPD
Phenol	625, 8270B	14.5-47.7	42	8270B	5-112	5-112	33
2-Chlorophenol	625, 8270B	41.3-109	40	8270B	23-134	23-134	24
4-Chloro-3-methylphenol	625, 8270B	56.6-115	31	8270B	22-147	22-147	23
4-Nitrophenol	625, 8270B	10.3-78	50	8270B	10-132	10-132	39
Pentachlorophenol	625, 8270B	51-133	27	8270B	14-176	14-176	17
1,4-Dichlorobenzene	625, 8270B	27.8-103	28	8270B	20-124	20-124	20
N-Nitroso-di-n-propylamine	625, 8270B	39.5-113	38	8270B	10-230	10-230	33
1,2,4-Trichlorobenzene	625, 8270B	36.1-104	28	8270B	44-142	44-142	23
Acenaphthene	625, 8270B	49.4-107	31	8270B	47-145	47-145	19
2,4-Dinitrotoluene	625, 8270B	48.5-118	41	8270B	39-139	39-139	21
Pyrene	625, 8270B	44.2-119	37	8270B	52-115	52-115	24

Table 10. MDL & MRL data for 8270B

	Water (ug/L)		Soil (mg/Kg)		
COMPOUND	MDL 3510	MRL	MDL 3545	MDL 3550	MRL
1,2,4-Trichlorobenzene	0.204	5.0	0.058	0.06	0.20
1,2-Dichlorobenzene	0.372	5.0	0.057	0.08	0.20
1,3-Dichlorobenzene	0.083	5.0	0.039	0.14	0.20
1,4-Dichlorobenzene	0.291	5.0	0.069	0.10	0.20
2,4,5-Trichlorophenol	0.138	5.0	0.049	0.08	0.20
2,4,6-Trichlorophenol	0.387	5.0	0.057	0.06	0.20
2,4-Dichlorophenol	0.203	5.0	0.074	0.07	0.20
2,4-Dimethylphenol	0.361	5.0	0.057	0.11	0.20
2,4-Dinitrophenol	0.415	25.0	0.041	0.06	1.00
2,4-Dintrotoluene	0.185	5.0	0.060	0.03	0.20
2,6-Dinitrotoluene	0.234	5.0	0.077	0.11	0.20
2-Chloronaphthalene	0.224	5.0	0.057	0.07	0.20
2-Chlorophenol	0.208	5.0	0.078	0.10	0.20
2-Methyl-4,6-dinitrophenol	0.155	25.0	0.083	0.06	1.00
2-Methylnaphthalene	0.238	5.0	0.068	0.05	0.20
2-Methylphenol	0.232	5.0	0.070	0.10	0.20
2-Nitroaniline	0.216	5.0	0.036	0.06	0.20
2-Nitrophenol	0.256	5.0	0.078	0.10	0.20
3,3'-Dichlorobenzidine	0.267	25.0	0.400	0.29	1.00
3-Nitroaniline	0.320	5.0	0.046	0.19	0.20
4-Bromophenyl phenyl ether	0.157	5.0	0.061	0.08	0.20
4-Chloro-3-methylphenol	0.151	5.0	0.079	0.07	0.20
4-Chloroaniline	0.185	5.0	0.571	0.06	1.00
4-Chlorophenyl phenyl	0.204	5.0	0.054	0.08	0.20
ether	0.262	5.0	0.076	0.05	0.20
4-Methylphenol	0.121	5.0	0.047	0.06	0.20
4-Nitroaniline	0.206	5.0	0.119	0.11	0.20
4-Nitrophenol	0.261	5.0	0.056	0.16	0.20
Acenaphthene	0.248	5.0	0.052	0.09	0.20
Acenaphthylene	0.140	5.0	0.049	0.04	0.20
Anthracene		5.0			0.20
Azobenzene		25.0			1.00
Benzidine	0.128	5.0	0.061	0.05	0.20
Benzo(a)anthracene	0.098	5.0	0.061	0.07	0.20
Benzo(a)pyrene	0.068	5.0	0.079	0.08	0.20
Benzo(b)fluoranthene	0.042	5.0	0.049	0.05	0.20
Benzo(g,h,i)perylene	0.151	5.0	0.058	0.07	0.20
Benzo(k)fluoranthene	0.288	5.0	0.079	0.11	0.20
Benzyl alcohol	0.379	5.0	0.073	0.09	0.20
Bis(2-chloroethoxy)methane	0.329	5.0	0.058	0.12	0.20
Bis(2-chloroethylether)	0.340	5.0	0.050	0.10	0.20
Bis(2-	0.311	5.0	0.062	0.11	0.20
chloroisopropyl)ether	0.239	5.0	0.056	0.08	0.20
Bis(2-ethylhexyl)phthalate	0.172	5.0	0.050	0.08	0.20
Butyl benzyl phthalate	0.145	5.0	0.076	0.04	0.20
Carbazole	0.138	5.0	0.034	0.15	0.20
Chrysene	0.120	5.0	0.041	0.14	0.20
Di-n-butylphthalate	0.113	5.0	0.046	0.10	0.20

Di-n-octylphthalate	0.216	5.0	0.042	0.08	0.20
Dibenzo(a,h)anthracene	0.149	5.0	0.058	0.09	0.20
Dibenzofuran	0.209	5.0	0.044	0.08	0.20
Diethylphthalate	0.188	5.0	0.057	0.10	0.20
Dimethylphthalate	0.090	5.0	0.057	0.07	0.20
Diphenylamine	0.218	5.0	0.057	0.09	0.20
Fluoranthene	0.179	5.0	0.053	0.10	0.20
Fluorene	0.264	5.0	0.059	0.09	0.20
Hexachlorobenzene	0.308	5.0	0.043	0.09	0.20
Hexachlorobutadiene	0.311	5.0	0.022	0.11	0.20
Hexachlorocyclopentadiene	0.127	5.0	0.029	0.06	0.20
Hexachloroethane	0.283	5.0	0.081	0.06	0.20
Indeno(1,2,3 cd)pyrene	0.237	25.0	0.125	0.08	1.00
Isophorone	0.240	25.0	0.070	0.11	1.00
N-Nitrosodimethylamine	0.329	5.0	0.066	0.07	0.20
N-Nitrosodipropylamine	0.300	5.0	0.061	0.09	0.20
Naphthalene	0.183	5.0	0.043	0.06	0.20
Nitrobenzene	0.135	5.0	0.039	0.06	0.20
Pentachlorophenol	0.087	5.0	0.073	0.10	0.20
Phenanthrene	0.217	5.0	0.061	0.08	0.20
Phenol		5.0	0.07		0.20
Pyrene					
Pyridine					

Attachment QAPP-B6

Organochlorine Pesticides and Chlorinated Polybiphenyls by GC/ECD Method 8081

GAM 8081

Organochlorine Pesticides and Polychlorinated Biphenyls by GC/ECD
Revision 2.0 2/05/02

1.0 SCOPE AND APPLICATION

1.1 Method 8081 is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). The following compounds can be determined by this method:

ANALYTE:	CAS. No.(a)
Aldrin	309-00-2
alpha-BHC	319-84-6
beta-BHC	319-85-7
delta-BHC	319-86-8
gamma-BHC (Lindane)	58-89-9
Chlordane (Technical)	12789-03-6
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33212-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
4,4'-Methoxychlor	72-43-5
Toxaphene	8001-35-2
Aroclor-1016	12674-11-2
Aroclor-1221	1104-28-2
Aroclor-1232	11141-16-5
Aroclor-1242	53469-21-9
Aroclor-1248	12672-29-6
Aroclor-1254	11097-69-1
Aroclor-1260	11096-82-5

(a) Chemical Abstract Services Registry Number

INSTRUMENTATION: GC

1.2 Table 1 lists the matrix specific method detection and reporting limits for each compound. Table 2 lists the estimated quantitation limit (EQL) for other matrices.

2.0 SUMMARY OF METHOD

2.1 Method 8081 provides gas chromatographic conditions for the detection of ppb concentrations of certain organochlorine pesticides and PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used (GAM 3550 for soils, GAM 3510 for waters).

2.2 The sensitivity of Method 8081 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8081 may also be performed on

samples that have undergone cleanup. GCM 3620A Florisil Column Cleanup and/or GCM 3665A Sulfuric Acid/Permanganate Cleanup (PCB's only) may be used to eliminate interferences in the analysis.

3.0 INTERFERENCES

3.1 Interferences by phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large late-eluting peaks. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatographs

4.1.1 Gas Chromatographs:

4.1.1.1 Varian 3400 CX with 8200 autosampler. Data is processed using HP 3365 Chemstation. Dual column system columns V1 and V2 below split from the same injection port.

4.1.1.2 Hewlett Packard 5890 with 7673A autosampler. Single column system for PCB's.

4.1.2 Columns

4.1.2.1 Column V1: Supelco PTE-5 30m x 0.25 mm x 0.25 µm.

4.1.2.2 Column V2: Supelco SPB-608 30m x 0.25 mm x 0.25 µm.

4.1.2.3 Column HP1: Restek RTx-5 30m x 0.53 mm x 1.5 µm.

4.1.3 Detectors: Electron capture (ECD) using nitrogen as make-up gas.

4.2 Concentrator apparatus: Zymark TurboVap Evaporator with 200 mL concentrator tubes.

4.3 Balances: Analytical, 0.0001 g and Top loading, 0.01 g.

4.4 Boiling chips: Solvent extracted, approximately 10/40 mesh, Teflon.

4.5 Volumetric flasks, Class A: sizes as appropriate with ground-glass stoppers.

4.6 Microsyringe: 10 uL.

4.7 Syringe: 1 mL.

4.8 Vials: Glass, 2, 10, and 40 mL capacity with Teflon-lined caps.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water.

5.3 Solvents

5.3.1 Hexane - Pesticide quality or equivalent.

5.3.2 Acetone - Pesticide quality or equivalent.
5.3.3 Toluene - Pesticide quality or equivalent.
5.3.4 Isooctane - Pesticide quality or equivalent.
5.3.5 Methanol - Pesticide quality or equivalent.
5.3.6 Ethyl Ether - Pesticide quality or equivalent preserved with 2% ethanol.

5.4 Stock standard solutions:

5.4.1 Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source:
Pesticide Mix A, Restek cat. no. 32003. 8-80 ug/ml.
Pesticide Mix B, Restek cat. no. 32004. 8-16 ug/ml.
Pesticide Surrogate Mix, Restek cat. no. 32000. 200 ug/ml.
Pesticide Matrix Spike Mix, Restek cat. no. 32018. 25-50 ug/ml.
Pesticide Performance Evaluation Mix, Restek cat.no.32002. 1-25 ug/ml
Aroclor Mixes, Restek cat. no. 32090. 1000 ug/ml.
Technical Chlordane Mix, Restek cat. no. 32021. 1000 ug/ml.
Toxaphene Mix, Restek cat. no. 32005. 1000 ug/ml.
note: standards equivalent to those above may also be used.

5.4.2 Stock standards for daily calibration checks are referenced in Section 5.4.1, but are of a different lot from which the calibration curve is made.

5.4.3 Transfer the stock standard solutions into vials with Teflon-lined screw caps or crimp tops. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.4.4 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.

5.5 Calibration standards: Calibration standards at a minimum of five concentrations for each parameter of interest are prepared through dilution of the stock standards with hexane. One of the concentrations should be at a concentration near, but above, the method detection limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after twelve months, or sooner, if comparison with check standards indicates a problem. Typical concentrations are 0.005-0.05, 0.010-0.10 0.020-0.20, 0.040-0.40, 0.060-0.60, 0.080-0.80 ug/mL (the concentrations for pesticides are analyte dependent, see Table 6) for pesticides, and 0.1, 0.5, 1.0, 1.5, 2.0, and 4.0 for PCB's, toxaphene, and chlordane.

5.6 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and organic-free reagent water blank with pesticide surrogates. Because GC/ECD data are much more subject to interference than GC/MS, a secondary surrogate is to be used when sample interference is apparent. Two surrogate standards, tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB), are added to each sample; however, only one need be calculated for recovery.

Proceed with corrective action when both surrogates are out of limits for a sample (Section 8.4).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Water and soil samples are stored refrigerated at 4°C. Water samples must be extracted within seven (7) days and soil samples extracted within 14 days of sample collection.

6.2 Extracts must be stored under refrigeration and must be analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1 Refer to the sample preparation section in this manual for guidance on choosing the appropriate extraction procedure. After addition of 1 mL of surrogate standards, water samples are extracted at a neutral, or as is, pH with methylene chloride, using GAM 3510B, and solid samples are extracted using GAM 3550A.

7.1.2 Prior to gas chromatographic analysis, the extraction solvent must be exchanged to hexane. The exchange is performed during the concentration procedures listed in all of the extraction methods. The exchange is performed as follows:

7.1.2.1 After concentrating to a 1 mL methylene chloride extract, a Teflon boiling chip is added, 10 mls of hexane are added to rinse the walls of the tube, and the temperature set to its highest setting (45 C). The extract is then blown down to 1 ml, 10 mls of hexane is added a 2nd time and taken to a final volume of 5ml for water samples, and 10mls for soil samples then placed in a 1.8ml autosampler vial. If cleanup is required, see GCM 3620A, otherwise proceed with gas chromatographic analysis.

7.2 Gas chromatographic conditions (Recommended):

7.2.1 Column V1: Supelco PTE-5 30m x 0.25 mm x 0.25 µm, or equivalent
Carrier gas (helium) flow rate: 3.0 mls/min (26 psi)
Make up gas (nitrogen) flow rate: 25 mls/min
Attenuation: 8 Range: 10
Temperature profile: 8 C/min.
Column:110(0)----------->290(7)
Injector: 240 Detector: 320

7.2.2 Column V2: Supelco SPB-608 30m x 0.25 mm x 0.25 µm or equivalent
Carrier gas (helium) flow rate: 3.0 mls/min (26 psi)
Make up gas (nitrogen) flow rate: 25 mls/min
Attenuation: 8 Range: 10
Temperature profile: 8 C/min.
Column:110(0)----------->290(7)
Injector: 240 Detector: 320

7.2.3 Column HP1: RTx-5 30 m x 0.53 mm, 1.5 µm or equivalent.
Carrier gas (helium) flow rate: 7.0 mls/min (53.1 cm/s)
Make up gas (nitrogen) flow rate: 25 mls/min
Attenuation: 8 Range: 10
Temperature profile: 15 10 C/min.
Column:60(2)----->150 (0)------>300(0)
Injector: 250 Detector: 300

7.3 Calibration:

7.3.1 The procedure for external standard calibration is used for gas chromatographic analyses. Calibration levels are listed in Section 5.5. Injection volume is 2 µL for all samples and standards. If cleanup is performed on the samples, the analyst should process a series of standards through the cleanup procedure and then analyze the samples by GC. This will validate elution patterns and the absence of interferents from the reagents.

7.3.2 Refer to section 8.3 for the procedure for establishing retention time windows for initial and continuing calibrations.

7.3.3 Assemble a calibration curve by running the standards under the chromatographic conditions used for the method. Use the Chemstation software to assemble an external standard curve based on the integrated areas of the peaks of interest.

7.3.4 External standard calibration procedure

7.3.4.1 For each analyte of interest, prepare calibration standards at a minimum of five concentrations by adding volumes of one or more stock standards to a vial and diluting to volume with an appropriate solvent. The %RSD for the five point curve of each analyte must be less than 20% for the curve to be valid.

7.3.4.1.1 Method 8081 has many multi component target analytes. The target analytes chosen for calibration should be limited to those specified in the project plan. Sites may specify either pesticides or PCB's. Multicomponent pesticides such as chlordane and toxaphene may not be specified in the project plan and therefore do not need a calibration requirement for these components. In the instances where unspecified PCB's are requested in the project plan, a mixture of Aroclors 1016 and 1260 will be used to construct the initial calibration curve since these two Aroclors contain all congeners present in all regulated Aroclors. In addition, a midpoint standard for all Aroclors will be included in each initial calibration so the analyst has a reference to the elution pattern and retention times on each column.

7.3.4.1.2 Calibration verification is made at the beginning of each 12 hour shift. All target analytes in the project plan must be injected with the exception of the Aroclors. Aroclor calibration verification need only include the Aroclor(s) present or if unknown a mid concentration mix of Aroclor 1016/1260 may be injected (Figure 3). However, if specific Aroclors are found during the initial screening, it is required that the samples containing those Aroclors be re-injected with the appropriate mid range Aroclor standard.

7.3.4.2 Because of the low concentration of pesticide standards injected on the GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore the GC column should be primed or deactivated by injecting a PCB or pesticide standard at a concentration 20 times more concentrated than the mid-concentration standard. Inject this standard prior to beginning the initial or continuing calibration verification. CAUTION : Several analytes, including Aldrin, may be observed in the injection just following this system priming. Always run an acceptable blank prior to running any standards or samples.

7.3.4.3 Calculate calibration factors for each analyte by injecting each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. Tabulate peak area responses against the amount injected in ug/ml. The results can be used to prepare a calibration curve for each analyte.

7.3.4.4 The working calibration curve or calibration factor must be verified at the beginning of each 12 hour shift, after every 20 samples and at the end of the run sequence by the injection of one calibration standard. If the response for any analyte varies from the predicted response by more than +/- 15%, a new calibration curve must be prepared for that analyte.

$$\text{Percent Difference (Drift)} = \frac{R1 - R2 \times 100}{R1}$$

where:

R1 = Calibration Factor from first analysis.
R2 = Calibration Factor from succeeding analyses.

7.3.4.5 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s)(each 12 hr. shift), or calibration standards interspersed within the samples. All samples that were injected after the standard that last met the QC criteria must be re-injected.

7.3.4.6 Although analysis of a single mid-concentration standard standard mixture or multi-component analyte) will satisfy the minimum requirements, analysts are urged to use different calibration verification standards during organochlorine pesticide/PCB analyses. Also, multi-level standards (mixtures or multi-component analytes) are highly recommended to ensure that detector response remains stable for all analytes over the calibration range.

7.3.5 Load the file for the lowest standard run, integrate the file to return the peak areas and retention times of the peaks in the chromatogram. Select "Prep/Recalibrate" to create a calibration table and select "New" to make a new table. This function loads any integrated peaks into a calibration table. Use the cursor to select any unwanted peaks and use the "Remove Peak" key to remove peaks that are not desired in the calibration table. The "Add peak" key may be used to introduce a new peak (analyte) to an existing calibration table. Prepare the first calibration level and enter the appropriate amount in nanograms. Repeat this process for each successive standard until all the standard concentration levels are entered. Peaks that are manually integrated or substituted during analysis must be verified and initialed by the QA officer.

7.3.6 After the calibration file is complete, save the method and the calibration file will be saved with the method file. Select "overwrite" if the method file already exists.

7.4 Gas chromatographic analysis:

7.4.2 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for more than a day. Therefore, the GC column should be primed or deactivated by injecting a high level PCB or pesticide standard. Inject this prior to beginning initial or daily calibration verification.

7.4.3 DDT and Endrin are easily degraded in the injection port if the injection port or front of the column is dirty. This is the result of buildup of high boiling residue from sample injection. Check for degradation problems at the beginning of each 12 hour shift by injecting a mid-concentration standard containing only 4,4'-DDT and Endrin, or Mix A. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and Endrin (Endrin ketone and Endrin aldehyde). If degradation of

either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration, by following the GC system maintenance outlined in Method 8000. Calculate percent breakdown as follows:

$$\text{\% breakdown for 4,4'-DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD)}}{\text{Total DDT peak areas (DDT + DDE + DDD)}} \times 100$$

$$\text{\% breakdown for Endrin} = \frac{\text{Total Endrin degradation peak area (Endrin aldehyde + Endrin ketone)}}{\text{Total Endrin peak areas (Endrin + Endrin aldehyde + Endrin ketone)}} \times 100$$

7.4.7 Using external calibration procedure, determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes.

7.4.8 Follow the instructions in Section 8.0 for instructions on the analyses sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a 0.020/0.040 ug/mL calibration check standard at the beginning, after each group of 10 samples and at the end of the analysis sequence.

7.4.9 Record the sample numbers and the order in which run in the sequence logbook for each run sequence. The resulting peak sizes in area units are saved by the data system software and processed using an automated integration/calibration package.

7.4.10 Using the external calibration procedure, determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes. See Section 7.6 for calculation equations.

7.4.11 If peak detection and identification are prevented due to interferences, the extract may undergo cleanup using GCM 3620.

7.4.12 Identification of mixtures (i.e. PCBs and Toxaphene) is based on the characteristic "fingerprint" retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peak(s) of the same retention time and shape generated using either internal or external calibration procedures.

7.4.13 Quantitation of the target compounds is based on: 1) a reproducible response of the ECD or ELCD within the calibration range; and 2) a direct proportionality between the magnitude of response of the detector to peaks in the sample extract and the calibration standards. Proper quantitation requires the appropriate selection of a baseline from which the area or height of the characteristic peak(s) can be determined.

7.4.14 If compound identification or quantitation are precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun sample on another instrument to

determine if the problem results from analytical hardware or the sample matrix.

7.5 Cleanup:

7.5.1 Proceed with GCM 3620A, followed by, if necessary, GCM 3665, using the 1 mL hexane extracts obtained from Section 7.1.2.1.

7.5.2 Following cleanup, the extracts should be analyzed by GC, as described in the previous sections.

7.5.3 If only PCBs are to be measured in a sample, the sulfuric acid/permanganate cleanup (GCM 3665), and/or Florisil Cleanup (GCM 3620A), is recommended.

7.6 Calculations

Parameter	Equation	Units
Water concentration	$\frac{(A_x)(A)(V_t)}{(A_s)(V_i)(V_s)} \times d$ where : A_x = peak area of the of the analyte being measured. A = amount of standard injected in nanograms (ng). d = dilution factor, dimensionless. V_i = volume injected in µL A_s = peak area of the external standard. V_t = final volume of extract in µL. V_s = volume of sample extracted in mL.	µg/L
Soil concentration (wet weight)	$\frac{(A_x)(A)(V_t)}{(A_s)(V_i)(W)(D_s)} \times d$ where : A_x = peak area of the of the analyte being measured. A = amount of standard injected in nanograms (ng). d = dilution factor, dimensionless. V_i = volume injected in µL. A_s = peak area of the external standard. V_t = final volume of extract in µL. W = wet weight of sample extracted in grams. D_s = 1 for wet weight, or S/100 where : S = % solids = $\frac{\text{dry weight of sampl}}{\text{wet weight of samp.}} \times 100$	

Quantitation of Multiple Component Analytes:

7.6.1 Multi-component analytes present problems in measurement. Suggestions are offered in the following sections for handing Toxaphene, Chlordane, PCB, DDT, and BHC.

7.6.2 Toxaphene: Toxaphene is manufactured by the chlorination of camphenes, whereas Strobane results from the chlorination of a mixture of camphenes and pinenes. Quantitative calculation of Toxaphene or Strobane is difficult, but reasonable accuracy can be obtained. To calculate toxaphene on GC/ECD: (a) adjust the sample size so that the major Toxaphene peaks are 10-70% of full-scale deflection (FSD); (b) inject a Toxaphene standard that is estimated to be within +/-10 ng of the sample; (c) quantitate using the five major peaks or the total area of the Toxaphene pattern.

7.6.2.1 To measure total area, construct the baseline of standard Toxaphene between its extremities; and construct the baseline under the sample, using the distances of the peak troughs to baseline on the standard as a guide. This procedure is made difficult by the fact that the relative heights and widths of the peaks in the sample will probably not be identical to the standard.

7.6.2.2 A series of Toxaphene residues have been calculated using the total peak area for comparison to the standard and also using the area of the last four peaks only, in both sample and standard. The agreement between the results obtained by the two methods justifies the use of the latter method for calculating Toxaphene in a sample where the early eluting portion of the Toxaphene chromatogram shows interferences from other substances such as DDT.

7.6.3 Chlordane is a technical mixture of at least 11 major components and 30 or more minor components. Trans- and cis-Chlordane (alpha and gamma), respectively, are the two major components of technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.

7.6.3.1 The GC pattern of a chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of: constituents from the technical Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

7.6.3.2 Whenever possible, when Chlordane residue does not resemble technical Chlordane, the analyst should quantitate the peaks of alpha-Chlordane, gamma-Chlordane, and heptachlor separately against the appropriate reference materials, and report the individual residues.

7.6.3.3 When the GC pattern of the residue resembles that of technical Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using the five major peaks or the total area. If the heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation of the residue. If heptachlor and/or heptachlor

epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected chlordane area. (Note that octachloro epoxide, a metabolite of Chlordane, can easily be mistaken for heptachlor epoxide on a nonpolar GC column.)

7.6.3.4 To measure the total area of the Chlordane chromatogram, inject an amount of technical chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms.

7.6.4 Polychlorinated biphenyls (PCBs): Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of Toxaphene, Strobane, and Chlordane. In each case, the chemical is made up of numerous compounds which generate multi-peak chromatograms Also, in each case, the chromatogram of the residue may not match that of the standard.

7.6.4.1 Mixtures of PCBs of various chlorine contents were sold for many years in the U.S. by the Monsanto Co. under the trade name Aroclor (1200 series and 1016). Although these Aroclors are no longer marketed, the PCBs remain in the environment and are sometimes found as residues in foods, especially fish. The Aroclors most commonly found in the environment are 1242, 1254, and 1260.

7.6.4.2 PCB residues are generally quantitated by comparison to the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

7.6.4.3 PCB Quantitation - Quantitate the PCB residues by comparing the responses of 3 to 6 major peaks in each appropriate Aroclor standard with the peaks obtained from the chlorinated biphenyls in the sample extract. The amount of Aroclor is calculated using an individual response factor for each of the major peaks. The results of the 3 to 6 determinations are averaged. Major peaks are defined as those peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. Late eluting Aroclor peaks are generally the most stable in the environment.

7.6.4.4 When samples appear to contain weathered PCBs, treated PCBs or mixtures of Aroclors, use of Aroclor standards is not appropriate. Several diagnostic peaks useful for identifying non-Aroclor PCBs are identified in Table 8. Analysts should examine chromatographs containing these peaks carefully, as these samples may contain PCBs. PCB concentrations may be estimated from specific congeners by adding the concentration of the congener peaks listed in Table 9. The congeners are analyzed as single components. This approach will provide reasonable accuracy for Aroclors 1016, 1232, 1242 and 1248 but will underestimate the concentrations of Aroclors 1254, 1260 and 1221. It is highly recommended that heavily weathered, treated or mixed Aroclors be analyzed using GC/MS if concentration permits.

7.6.5 Hexachlorocyclohexane (BHC, from the former name, benzene hexachloride): Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor; it consists of a mixture of six chemically distinct isomers and one or more heptachlorocyclohexanes and octachlorocyclohexanes. Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. Quantitate each isomer (alpha, beta, gamma, and delta) separately against a standard of the respective pure isomer.

7.6.6 DDT: Technical DDT consists primarily of a mixture of 4,4'-DDT approximately 75%) and 2,4'-DDT (approximately 25%). As DDT weathers, 4,4'-DDE, 2,4'-DDE, 4,4'-DDD, and 2,4'-DDD are formed. Since the 4,4'-isomers of DDT, DDE, and DDD predominate in the environment, these are the isomers normally regulated by US EPA and should be quantitated against standards of the respective pure isomer.

7.6.7 Technical chlordane and toxaphene are pesticide mixtures containing a minimum of 10 components and up to 100. PCB's were manufactured as Aroclors, containing a large number of PCB's. Proper identification of these mixtures requires GC operators experienced in ECD chromatography and an understanding of how these compounds degrade over time. For example, Aroclor 1260 in the environment may only match the standard in the later portions of the chromatogram due to photolysis of the less chlorinated components, or a technical chlordane sample may have different peak area ratios due to metabolic processes or even because it was manufactured from a different company. These factors need to be carefully considered during identification. For these analytes, 6 prominent peaks are chosen for quantifying. These peaks are chosen based on their abundance in the standard, the ability to be easily resolved, and their inability to be mis-identified. For example, the late eluting peaks of Aroclor 1260 do not overlap Aroclor 1254. External calibration curves are made for each of these as if they were single component analytes. The peak names identify the Aroclor and the peak #, for example AR1254-2 would represent the second peak identified in Aroclor 1254. When quantifying, an average of concentrations is calculated and reported. In the case of interferences or degradation, an average of only those peaks with similar concentrations is calculated. For example, if the concentrations were calculated to be: AR1254-1=1.25 ug/l, AR1254-2=2.79 ug/l, AR1254-3=1.37 ug/l, AR1254-4=1.45 ug/l, AR1254-5=12.32 ug/l, and AR1254-6=0.45 ug/l; Peak 5 would be assumed high due to interference, and Peak 6 low due to degradation. Peaks 5 and 6 would not be used in to quantitate, and the reported value is calculated from the other four peaks : 1.72 ug/l.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent.

8.1.1 Calculate the values for the preparation blanks, laboratory control samples (LCS), matrix spikes(MS) and matrix spike duplicates(MSD)

8.1.1.1 If the preparation blank shows no contamination above the reporting limits for the analytes of interest, the method is presumed in control and sample analysis can proceed.

8.1.1.2 If the preparation blank contains contamination above the reporting limit, corrective actions must be performed to bring the method back into control. After the corrective actions are performed the analyst(s) must demonstrate that the preparation and analysis procedures are free of contaminants before sample analysis can proceed.

8.1.1.3 Calculate the spike recoveries for the LCS, MS and MSD. If all recoveries are within the established limits in Table 5. The method is presumed in control and sample analysis can proceed.

8.1.1.4 If the spike recoveries for the LCS are within the established control limits in Table 5, but the MS(and/or MSD) are not within the established limits in Table 5, the method is presumed in control and sample analysis can proceed. Sample data for the spiked sample with recoveries outside of the acceptance limits in Table 5. should be flagged as "estimated concentration."

8.1.1.5 If the spike recoveries for the LCS are not within the established control limits in Table 5, corrective actions must be performed to bring the method back into control. After corrective actions are performed, the analyst(s) must demonstrate LCS recoveries within the established limits before sample analysis can proceed.

8.2 Gas Chromatographic System Check

8.2.1 The quality control check sample concentrate should contain each single-component parameter of interest at the following concentrations in acetone or other water miscible solvent: g-BHC, Heptachlor, and Aldrin at 0.2 ug/ml; DDT, Endrin, and Dieldrin at 0.4 ug/ml. If this method is only to be used to analyze for PCBs, chlordane, or toxaphene, the QC check sample concentrate should contain the most representative multi-component parameter at a concentration of 10 ug/ml in acetone.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for method 8081. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both tables should be used to evaluate the laboratory's ability to perform and generate acceptable data by this method.

8.3 Retention time windows(also refer to GQAP 003)

8.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of all single component standard mixtures and multi-response products (i.e. PCBs) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight. Current retention times are listed in Table 1.

8.3.2 Calculate the standard deviation of the three absolute retention times (use any function of retention time; including absolute retention time, or relative retention time) for each single component standard. For multi-response products, choose one major peak from the envelope and calculate the standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in samples.

8.3.2.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. For multi-response analytes (i.e. PCBs), the analyst should use the retention time window, but should primarily rely on pattern recognition.

8.3.2.2 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard

deviation of a close eluting, similar compound to develop a valid retention time window.

8.3.3 The analyst must calculate retention time windows for each analyte on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

8.4 Corrective Actions

8.4.1 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within laboratory established limits. If surrogate recovery for both surrogates is not within limits, the following procedures are required.

8.4.1.1 Check to be sure there are no errors in calculations, surrogate solutions. Also, check instrument performance. You may use the criteria in section 8.2 above.

8.4.1.2 Recalculate the data and/or re analyze the extract if any of the above checks reveal a problem.

8.4.1.3 Re extract and re analyze the sample, if none of the above are a problem or flag the data as "estimated concentration."

8.4.1.4 Laboratory control samples and preparation blanks must have surrogate recoveries within laboratory established limits for the primary or secondary surrogate. If both surrogates are out of established limits proceed with corrective actions.

8.4.2 If the laboratory control sample (LCS) is out of control the following procedures are required.

8.4.2.1 Check to be sure there are no errors in calculations, spiking solutions. Also, check instrument performance. You may use the criteria in section 8.2 above.

8.4.2.2 Recalculate the data and/or re analyze the extract if any of the above checks reveal a problem.

8.4.2.3 Re-analyze the LCS sample to demonstrate that the analysis is in control.

8.4.2.4 Re-extract and re-analyze the LCS and all samples associated with the unacceptable LCS.

8.4.3 Samples that are prepared and run with an out of control preparation blank must be re-extracted and re-run along with a new preparation blank.

8.4.4 Flag data from samples that have unacceptable matrix spike recoveries or precision outliers as "estimated concentration".

9.0 METHOD PERFORMANCE :

The accuracy and precision obtained will be determined by the sample matrix, sample-preparation technique, optional cleanup techniques, and calibration procedures used.

10.0 REFERENCES

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10.9 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

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10.12 Stewart, J. "EPA Verification Experiment for Validation of the SOXTEC (R) PCB Extraction Procedure"; Oak Ridge National Laboratory, Oak Ridge, TN, 37831-6138; October 1988.

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TABLE 1. GAS CHROMATOGRAPHY OF PESTICIDES AND PCBs(a)

	Retention time (min)		Water		Soil	
Analyte	Col. V1	Col. V2	MDL (ug/L)	MRL (ug/L)	MDL (ug/kg)	MRL (ug/Kg)
Aldrin	13.59	15.29	0.010	0.05	0.745	1.7
Alpha-BHC	10.13	12.40	0.013	0.05	0.058	1.7
Beta-BHC	10.74	13.69	0.006	0.05	0.987	1.7
Delta-BHC	11.48	14.64	0.007	0.05	0.045	1.7
Gamma-BHC (Lindane)	10.59	13.44	0.005	0.05	0.370	1.7
Chlordane (technical)	e	e	0.050	2.50	8.173	85
4,4'-DDD	16.85	19.38	0.029	0.10	0.077	3.3
4,4'-DDE	15.89	18.06	0.014	0.10	0.260	3.3
4,4'-DDT	17.69	20.10	0.022	0.10	0.856	3.3
Dieldrin	15.97	18.30	0.015	0.10	0.110	3.3
Endosulfan I	15.36	17.57	0.010	0.05	0.062	1.7
Endosulfan II	16.67	19.57	0.025	0.10	0.111	3.3
Endosulfan sulfate	17.63	20.66	0.037	0.10	0.163	3.3
Endrin	16.47	19.12	0.009	0.10	0.255	3.3
Endrin aldehyde	21.02	21.10	0.054	0.10	0.150	3.3
Heptachlor	12.74	14.44	0.007	0.05	0.040	1.7
Heptachlor epoxide	14.15	16.67	0.007	0.05	0.680	1.7
Methoxychlor	22.79	21.97	0.062	0.50	14.12	17
Toxaphene	e	e	0.110	2.50	1.98	85
PCB-1016	e	e	0.145	1.50	6.23	33
PCB-1221	e	e	0.039	1.50	4.81	33
PCB-1232	e	e	0.029	1.50	3.70	33
PCB-1242	e	e	0.145	1.50	6.23	33
PCB-1248	e	e	0.105	1.50	1.57	33
PCB-1254	e	e	0.135	1.50	3.35	33
PCB-1260	e	e	0.103	1.50	3.96	33
TCMX	9.16	10.47		1.50		33
DCB	28.98	25.75		1.50		33

(a)U.S. EPA. Method 617. Organochlorine Pesticides and PCBs. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

(e)= Multiple peak response. See Table 7 for retention times.

(nd)= not determined.

TABLE 2. DETERMINATION OF ESTIMATED QUANTITATION LIMITS (EQLs) FOR VARIOUS MATRICES(a)

Matrix	Factor(b)
Ground water	10
Low-concentration soil by sonication with GPC cleanup	670
High-concentration soil and sludges by sonication	10,000
Non-water miscible waste	100,000

(a) Sample EQLs are highly matrix-dependent. The EQLs listed herein are

provided for guidance and may not always be achievable.

(b) EQL = [Method detection limit (Table 1)] * [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

Table 3.
QC ACCEPTANCE CRITERIA(a)

Analyte	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{x}$ (ug/L)	Range P, P(s) (%)
Aldrin	2.0	0.42	1.08-2.24	42-122
Alpha-BHC	2.0	0.48	0.98-2.44	37-134
Beta-BHC	2.0	0.64	0.78-2.60	17-147
Delta-BHC	2.0	0.72	1.01-2.37	19-140
Gamma-BHC	2.0	0.46	0.86-2.32	32-127
Chlordane	50	10.0	27.6-54.3	45-119
4,4'-DDD	10	2.8	4.8-12.6	31-141
4,4'-DDE	2.0	0.55	1.08-2.6	30-145
4,4'-DDT	10	3.6	4.6-13.7	25-160
Dieldrin	2.0	0.76	1.15-2.49	36-146
Endosulfan I	2.0	0.49	1.14-2.82	45-153
Endosulfan II	10	6.1	2.2-17.1	D-202
Endosulfan Sulfate	10	2.7	3.8-13.2	26-144
Endrin	10	3.7	5.1-12.6	30-147
Heptachlor	2.0	0.40	0.86-2.00	34-111
Heptachlor epoxide	2.0	0.41	1.13-2.63	37-142
Toxaphene	50	12.7	27.8-55.6	41-126
PCB-1016	50	10.0	30.5-51.5	50-114
PCB-1221	50	24.4	22.1-75.2	15-178
PCB-1232	50	17.9	14.0-98.5	10-215
PCB-1242	50	12.2	24.8-69.6	39-150
PCB-1248	50	15.9	29.0-70.2	38-158
PCB-1254	50	13.8	22.2-57.9	29-131
PCB-1260	50	10.4	18.7-54.9	8-127

(s) = Standard deviation of four recovery measurements, in ug/L.

(x) = Average recovery for four recovery measurements, in ug/L.

(P), (P)(s) = Percent recovery measured.

(D) = Detected; result must be greater than zero.

(a)Criteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

Table 4.
METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION(a)

Analyte	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s(r)' (ug/L)	Overall precision, S' (ug/L)
Aldrin	0.81C+0.04	0.16x-0.04	0.20x-0.01
Alpha-BHC	0.84C+0.03	0.13x+0.04	0.23x-0.00
Beta-BHC	0.81C+0.07	0.22x+0.02	0.33x-0.95
Delta-BHC	0.81C+0.07	0.18x+0.09	0.25x+0.03
Gamma-BHC	0.82C-0.05	0.12x+0.06	0.22x+0.04
Chlordane	0.82C-0.04	0.13x+0.13	0.18x+0.18
4,4'-DDD	0.84C+0.30	0.20x-0.18	0.27x-0.14
4,4'-DDE	0.85C+0.14	0.13x+0.06	0.28x-0.09
4,4'-DDT	0.93C-0.13	0.17x+0.39	0.31x-0.21
Dieldrin	0.90C+0.02	0.12x+0.19	0.16x+0.16
Endosulfan I	0.97C+0.04	0.10x+0.07	0.18x+0.08
Endosulfan II	0.93C+0.34	0.41x-0.65	0.47x-0.20
Endosulfan Sulfate	0.89C-0.37	0.13x+0.33	0.24x+0.35
Endrin	0.89C-0.04	0.20x+0.25	0.24x+0.25
Heptachlor	0.69C+0.04	0.06x+0.13	0.16x+0.08
Heptachlor epoxide	0.89C+0.10	0.18x-0.11	0.25x-0.08
Toxaphene	0.80C+1.74	0.09x+3.20	0.20x+0.22
PCB-1016	0.81C+0.50	0.13x+0.15	0.15x+0.45
PCB-1221	0.96C+0.65	0.29x-0.76	0.35x-0.62
PCB-1232	0.91C+10.79	0.21x-1.93	0.31x+3.50
PCB-1242	0.91C+10.79	0.21x-1.93	0.31x+3.50
PCB-1248	0.91C+10.79	0.21x-1.93	0.31x+3.50
PCB-1254	0.91C+10.79	0.21x-1.93	0.31x+3.50
PCB-1260	0.91C+10.79	0.21x-1.93	0.31x+3.50

(x') = Expected recovery for one or more measurements of a sample containing concentration C, in ug/L.

(s(r)') = Expected single analyst standard deviation of measurements at an average concentration of x, in ug/L.

(S') = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in ug/L. C - True value for the concentration, in ug/L.

(x) = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

Table 5.
Matrix Spike and Laboratory Control Sample Recovery Data For
ORGANOCHLORINE PESTICIDES
by Gas Chromatographic Methods 608 and 8081

COMPOUNDS	Water Methods (EPA/SW846)	[illegible]	Water Precision RPD	Soil Methods (SW846)	Soil Accuracy, %R	Soil Precision RPD
Aldrin	608/8081	58-118	0 - 20	8081	35-129	0 - 14
gamma-BHC (Lindane)	608/8081	58-110	0 - 15	8081	31-123	0 - 27
4,4'-DDT	608/8081	58-126	0 - 27	8081	48-134	0 - 35
Dieldrin	608/8081	64-130	0 - 18	8081	55-134	0 - 31
Endrin	608/8081	57-139	0 - 21	8081	68-128	0 - 20
Heptachlor	608/8081	57-118	0 - 20	8081	33-125	0 - 14
Aroclor 1260	608/8081	72-119	0 - 50	8081	51-104	0 - 27
TCMX(Surrogate)	608/8081	11-151		8081	37-130	
DCB(Surrogate)	608/8081	12-149		8081	31-147	

TABLE 6.
Analyte Stock Solution Concentrations

Analyte	Stock Concentration (ug/ml)
Alpha-BHC	8
Gamma-BHC	8
Beta-BHC	8
Heptachlor	8
Delta-BHC	8
Aldrin	8
Heptachlor epoxide	8
Endosulfan I	8
4,4'-DDE	16
Dieldrin	16
Endrin	16
4,4'-DDD	16
Endosulfan II	16
4,4'-DDT	16
Endrin aldehyde	16
Endosulfan sulfate	16
Methoxychlor	80
Aroclor 1016	1000
Aroclor 1221	1000
Aroclor 1232	1000
Aroclor 1242	1000
Aroclor 1248	1000
Aroclor 1254	1000
Aroclor 1260	1000

Table 7.
Retention Times for Multi-component Pesticides and PCB's

Compound	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Peak 6
Chlordane	10.4	10.9	12.7	13.9	14.0	15.9
Toxaphene	15.6	16.7	17.5	17.7	18.2	18.7
Aroclor 1016	12.7	13.3	13.5	13.8	14.1	14.3
Aroclor 1221	9.86	11.3	11.6			

Aroclor 1232	11.6	12.7	13.1	13.3	14.1	14.3
Aroclor 1242	12.7	13.3	13.8	14.1	14.9	15.2
Aroclor 1248	13.8	14.1	14.3	14.8	14.9	15.2
Aroclor 1254	14.8	15.3	15.7	15.9	16.3	17.2
Aroclor 1260	16.3	16.7	17.2	17.5	17.9	18.8

Table 8.

PEAKS DIAGNOSTIC OF PCBs OBSERVED ON 30 meter X 0.53 mm ID RTX-5 COLUMN DURING SINGLE COLUMN ANALYSIS

Peak No. [a]	RT on RTX-5 [b]	Aroclor [c]
I	9.86	1221
II	11.3	1221, 1232, 1248
III	11.6	1016, 1221, 1232, 1242,
IV	12.7	1016, 1232, 1242, 1248,
V	13.3	1016, 1232, 1242,
VI	14.8	1248, 1254
VII	15.9	1254
VIII	16.3	1254
IX	17.2	1254, 1260
X	17.9	1260
XI	18.8	1260

[a] Peaks are sequentially numbered in elution order and are not isomer numbers.

[b] Column HP1: RTx-5, 30 m x 0.53 mm, 1.5 µm or equivalent.
Temperature program = 60 C for 2 minutes, then 15 C/min to 150 C hold 0 minutes, then 10 C/min to 300 C. Injector: 250. ECD Detector: 300

[c] Underline indicates largest peak in the pattern for that Aroclor.

Table 9.

SPECIFIC PCB CONGENERS IN AROCLORS

		Aroclor						
Congener	**IUPAC #**	**1016**	**1221**	**1232**	**1242**	**1248**	**1254**	**1260**
Biphenyl	--		X					
2-CB	1	X	X	X	X			
23-DCB	5	X	X	X	X	X		
34-DCB	12	X		X	X	X		
244'-TCB	28*	X		X	X	X	X	
22'35'-TCB	44			X	X	X	X	X
23'44'-TCB	66*					X	X	X
233'4'6-PCB	110						X	
23'44'5-PCB	118*						X	X
22'44'55'-HCB	153							X
22'344'5'-HCB	138							X
22'344'55'-HpCB	180							X
22'33'44'5-HpCB	170							X

*Apparent co-elution of: 28 with 31 (2,4',5-trichlorobiphenyl)
66 with 95 (2,2',3,5',6-pentachlorobiphenyl)
118 with 149 (2,2',3,4',5',6-hexachlorobiphenyl)

Figure 1.

Organochlorine Pesticides
Column V1

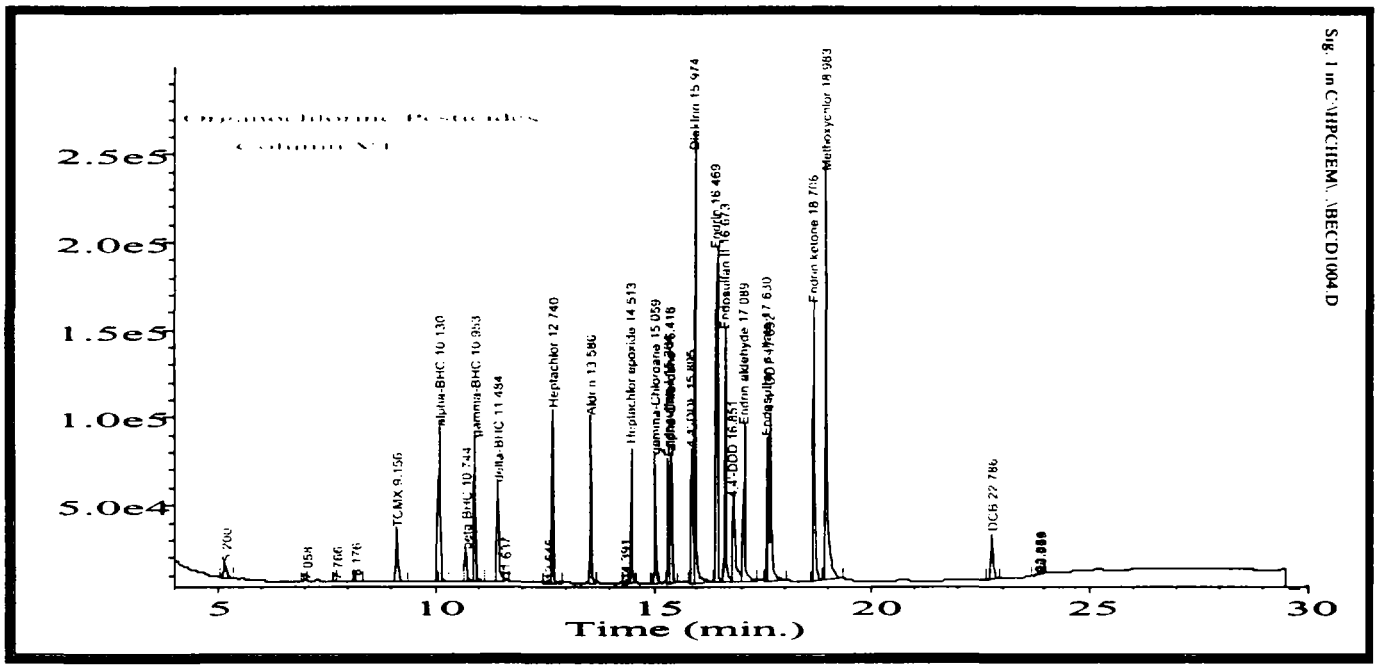


Figure 2.

Organochlorine Pesticides
Column V2

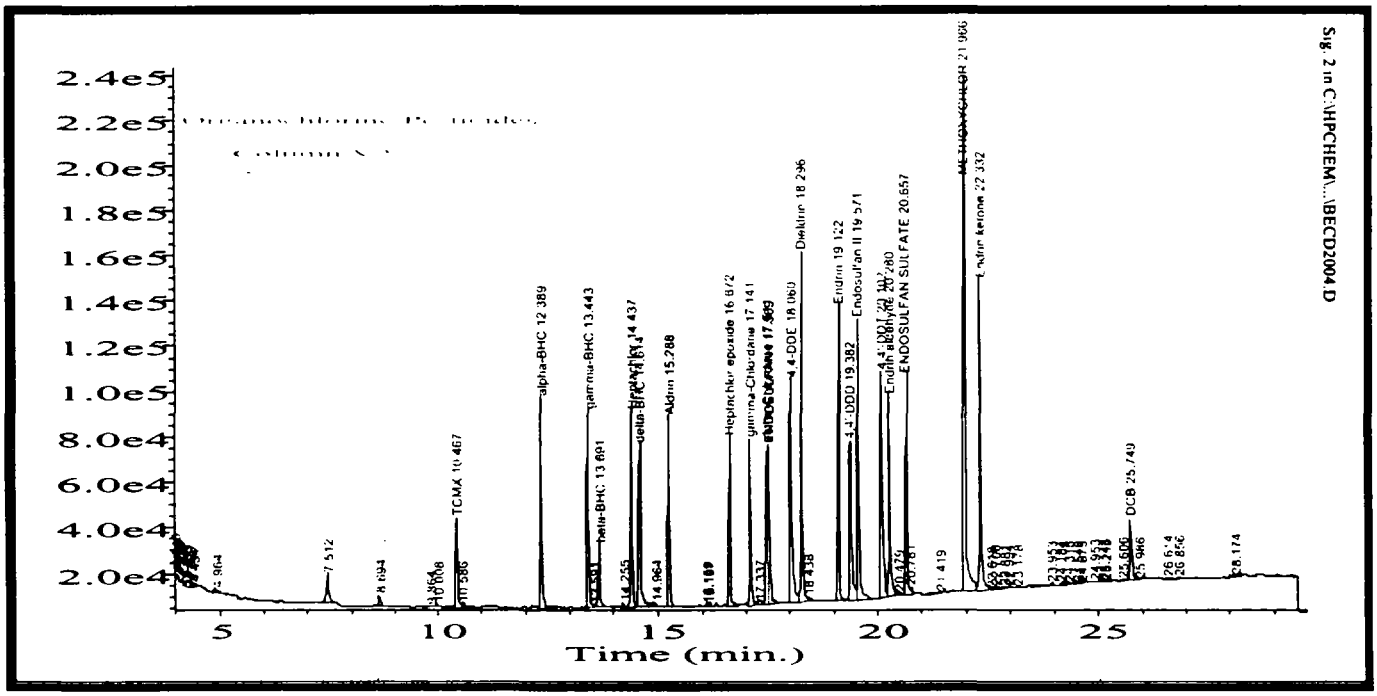


Figure 3

MIXED AROCLOR STANDARD

PCB 1016 and PCB 1260

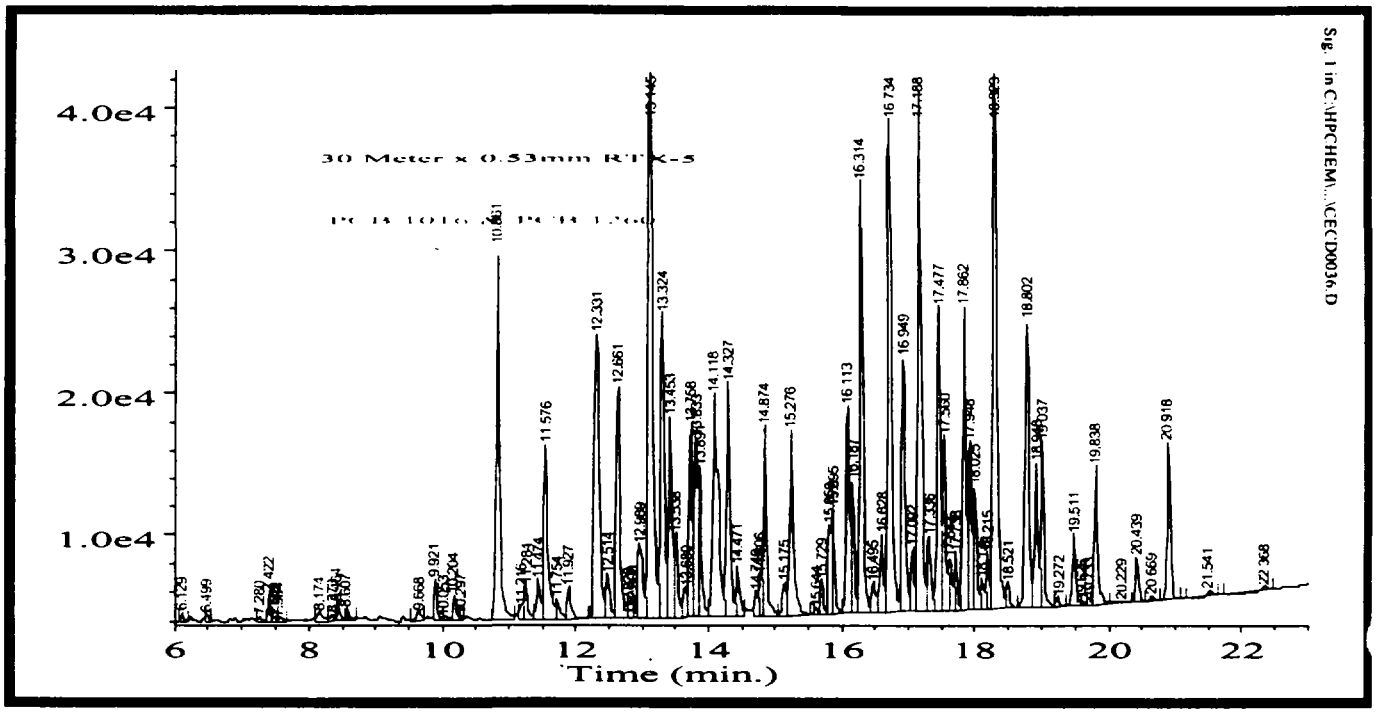


Figure 4

PCB 1016

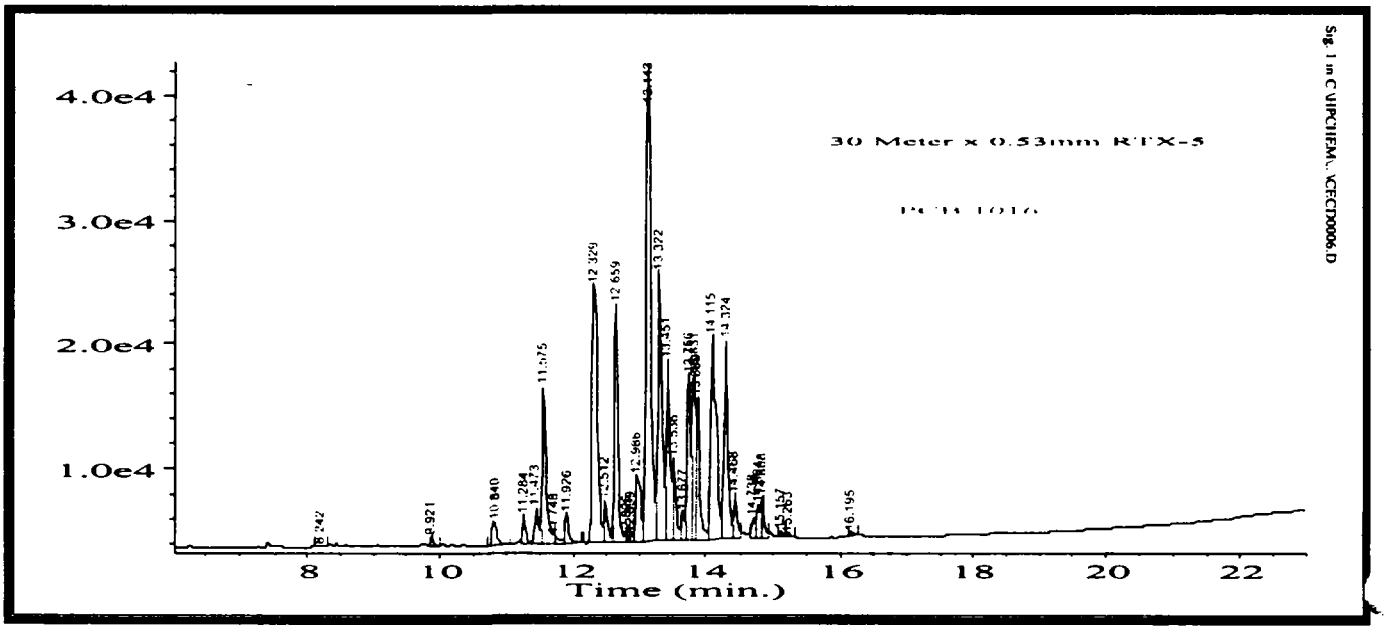


Figure 5.

PCB 1221

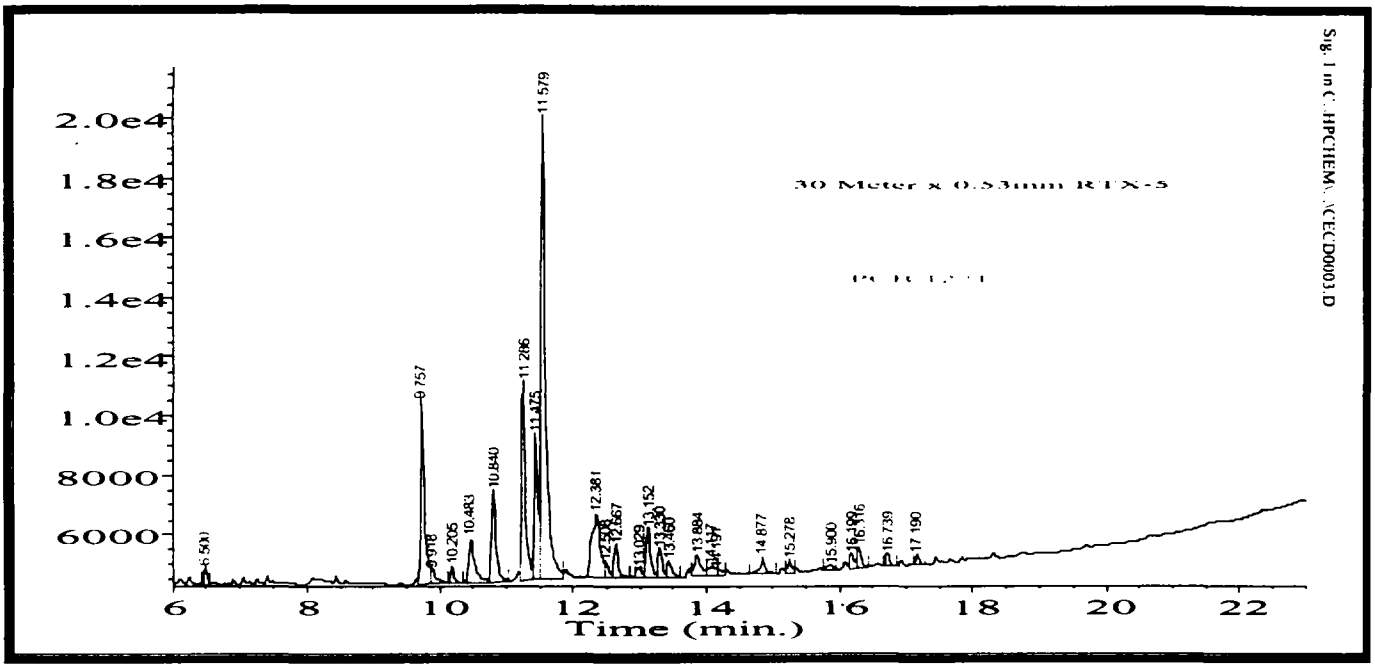


Figure 6.

PCB 1232

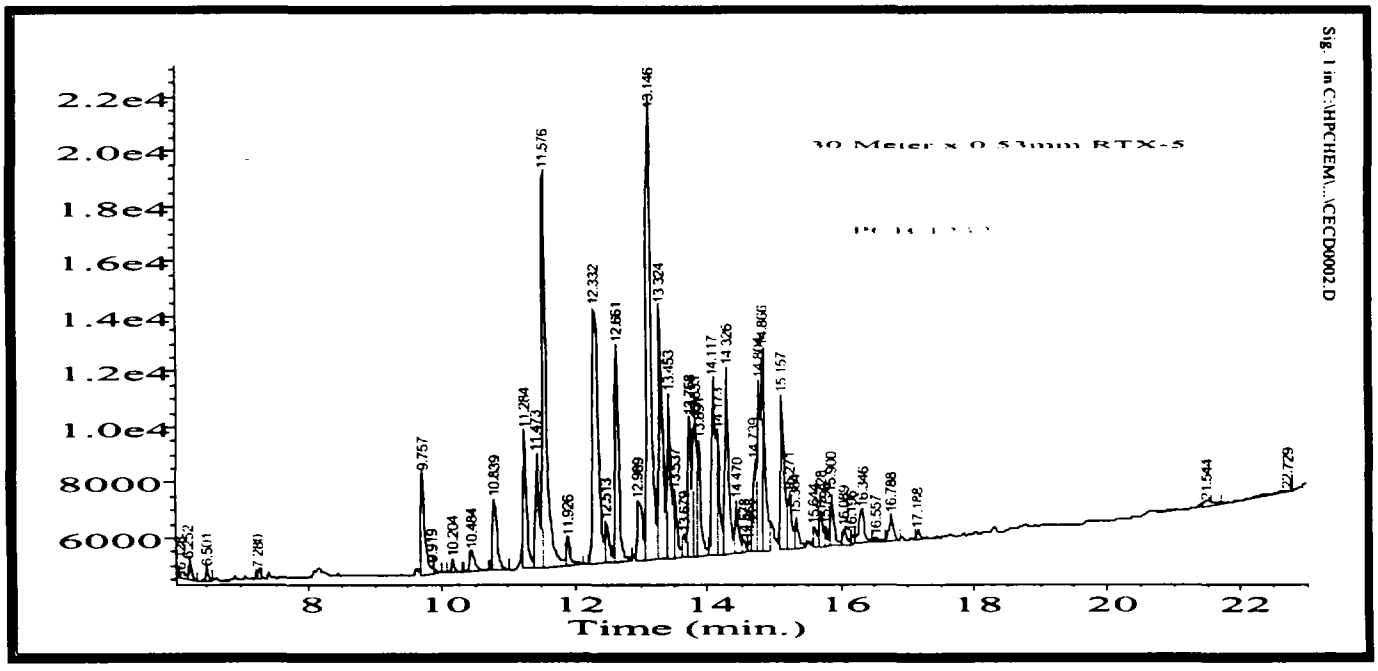


Figure 7.

PCB 1242

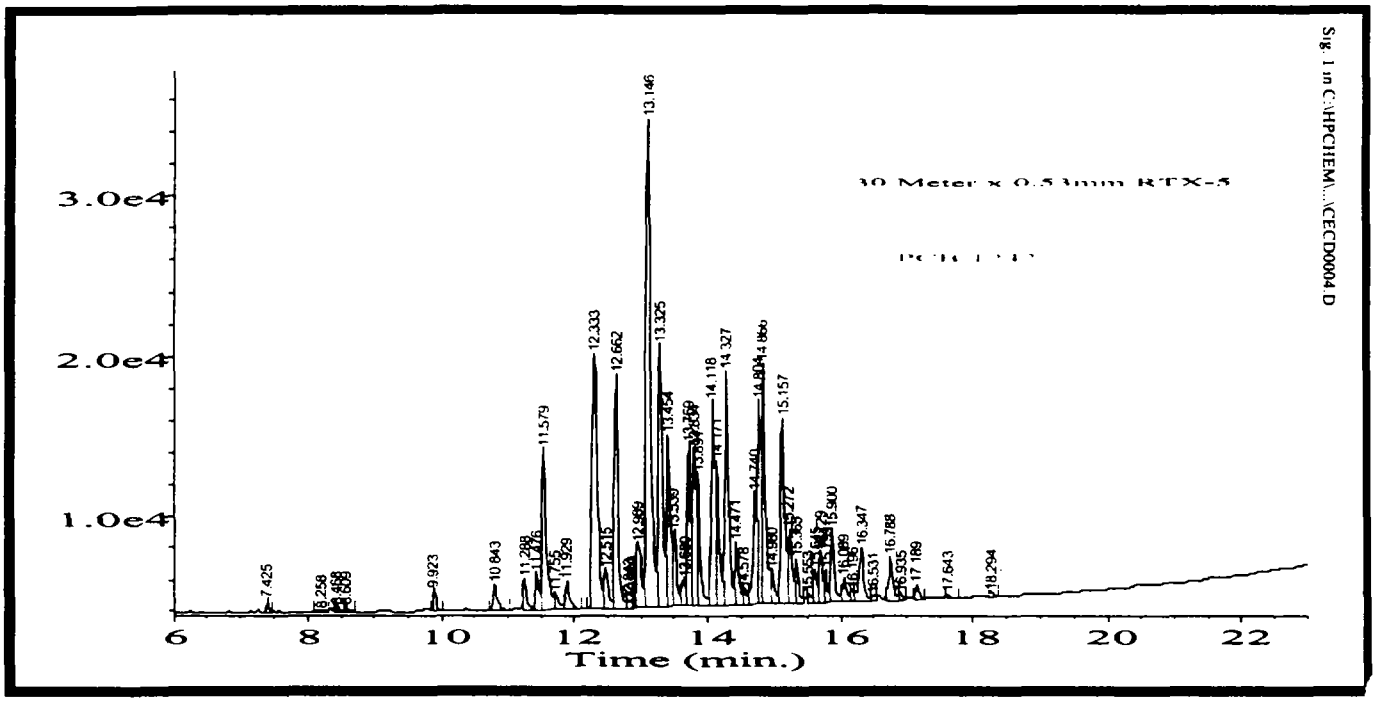


Figure 8.

PCB 1248

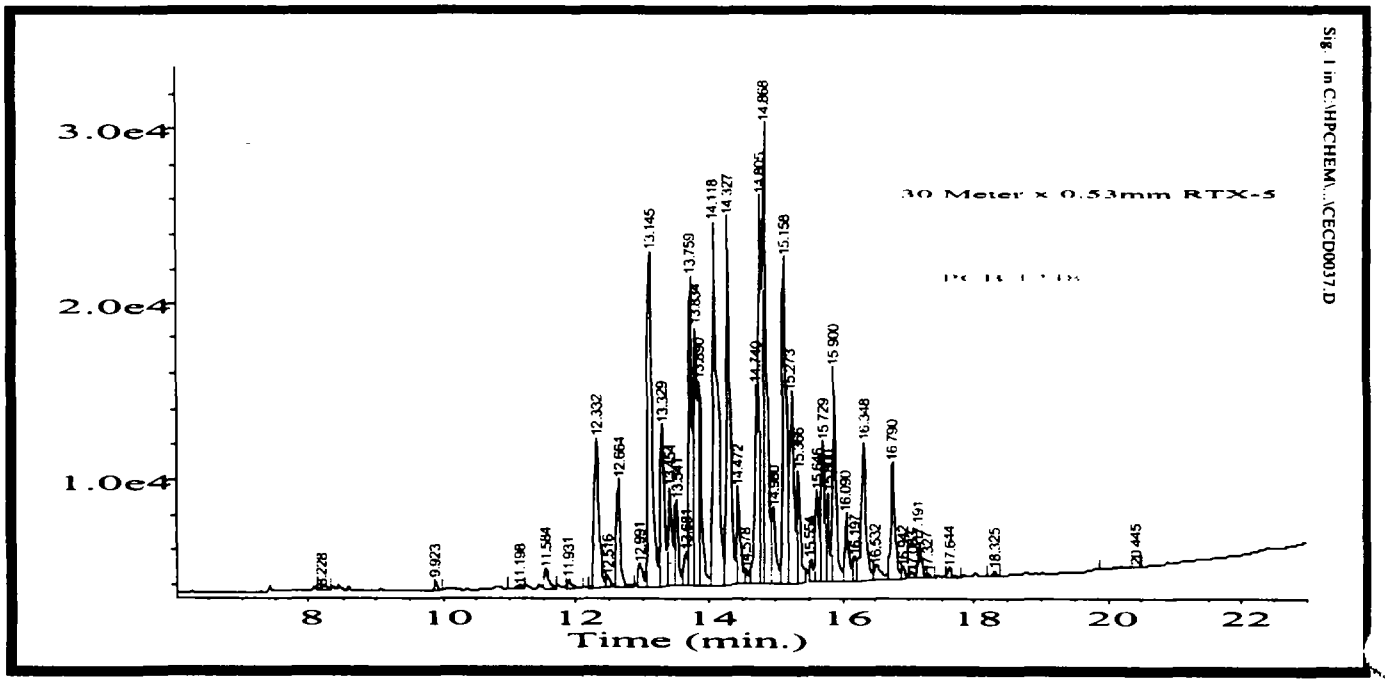


Figure 9.

PCB 1254

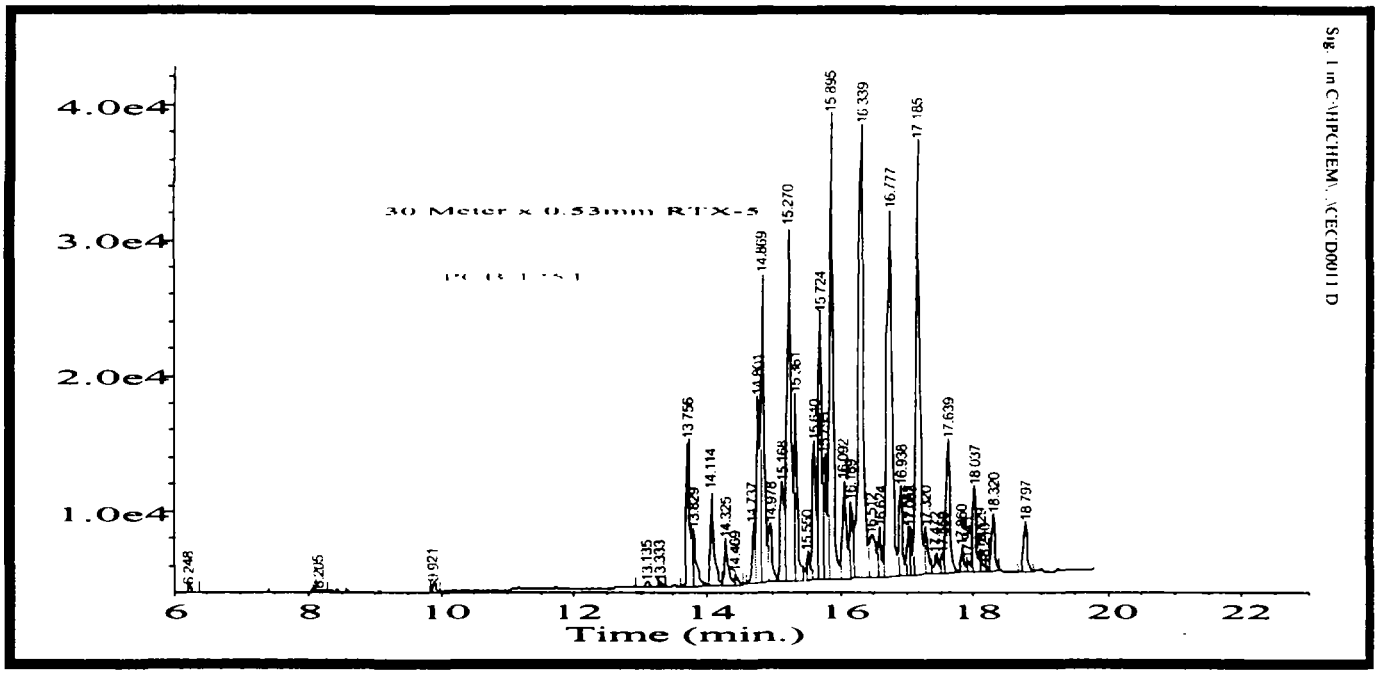
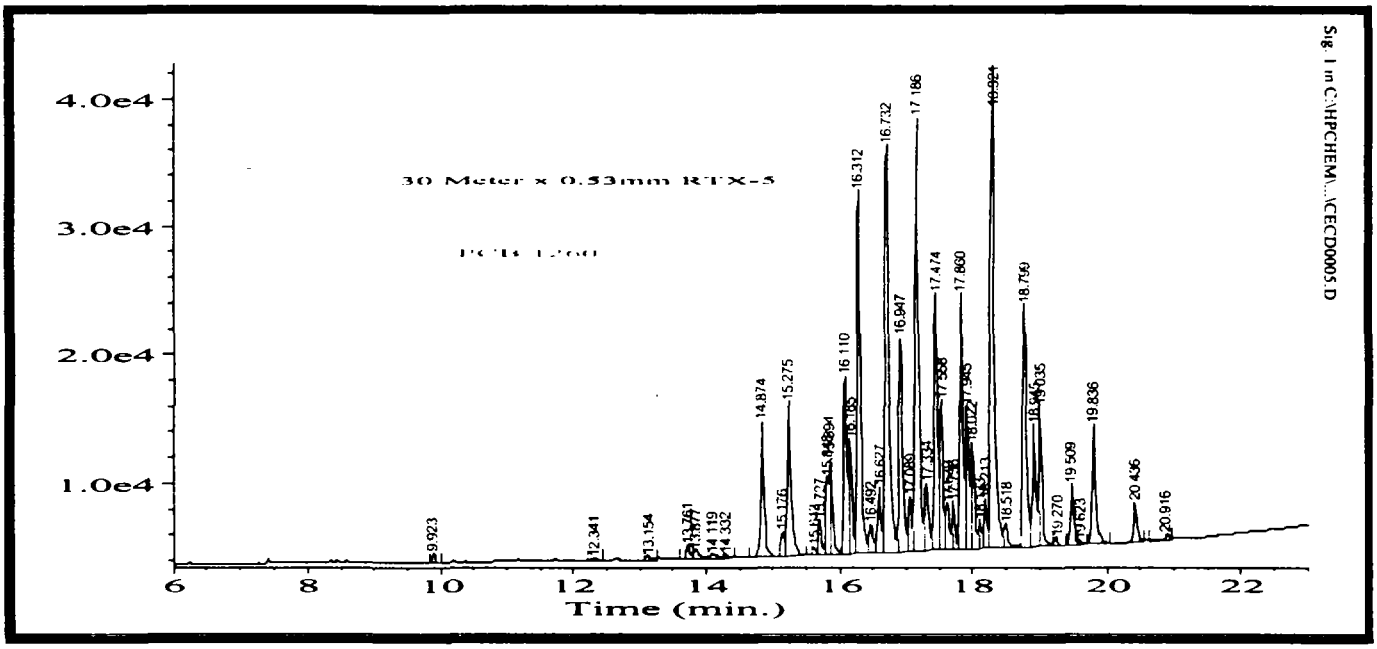


Figure 10.

PCB 1260



Attachment QAPP-B7

Toxicity Characteristic Leaching Procedure
Method 1311

GAM 1311
Toxicity Characteristic Leaching Procedure
Revision 3.0: 2/10/97

ANALYTES: (regulated compounds)

	CAS #	Regulatory level (mg/L)	HWNO•
Arsenic	7440-38-2	5.0	D004
Barium	7440-39-3	100.0	D005
Benzene*	71-43-2	0.5	D018
Cadmium	7440-43-9	1.0	D006
Carbon tetrachloride*	56-23-5	0.5	D019
Chlordane	57-74-9	0.03	D020
Chlorobenzene*	108-90-7	100.0	D021
Chloroform*	67-66-3	6.0	D022
Chromium	7440-47-3	5.0	D007
o-Cresol	95-48-7	200.0	D023
m-Cresol	108-39-4	200.0	D024
p-Cresol	106-44-5	200.0	D025
Cresol	--------	200.0	D026
2,4-D	94-75-7	10.0	D016
1,4-Dichlorobenzene	106-46-7	7.5	D027
1,2-Dichloroethane*	107-06-2	0.5	D028
1,1-Dichloroethylene*	75-35-4	0.7	D029
2,4-Dinitrotoluene	121-14-2	0.13	D030
Endrin	72-20-8	0.02	D012
Heptachlor (and its epoxide)	76-44-8	0.008	D031
Hexachlorobenzene	118-74-1	0.13	D032
Hexachlorobutadiene	87-68-3	0.5	D033
Hexachloroethane	67-72-1	3.0	D034
Lead	7439-92-1	5.0	D008
Lindane	58-89-9	0.4	D013
Mercury	7439-97-6	0.2	D009
Methoxychlor	72-43-5	10.0	D014
Methyl ethyl ketone*	78-93-3	200.0	D035
Nitrobenzene	98-95-3	2.0	D036
Pentachlorophenol	87-86-5	100.0	D037
Pyridine	110-86-1	5.0	D038
Selenium	7782-49-2	1.0	D010
Silver	7440-22-4	5.0	D011
Tetrachloroethylene*	127-18-4	0.7	D039
Toxaphene	8001-35-2	0.5	D015
Trichloroethylene*	79-01-6	0.5	D040
2,4,5-Trichlorophenol	95-95-4	400.0	D041
2,4,6-Trichlorophenol	88-06-2	2.0	D042
2,4,5-TP (Silvex)	93-72-1	1.0	D017
Vinyl chloride*	75-01-4	0.2	D043

• EPA Hazardous Waste Code
* volatiles
notes: Regulated analytes and regulatory levels from 40 CFR.
Additional analytes may be run per client request.

INSTRUMENTATION: Refer to appropriate analytical method.

1.0 SCOPE AND APPLICATION

1.0 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run (see table 1).

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of an extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.7µ glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see ANALYTES for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.7µ glass fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: Environmental Express 6 place agitation apparatus. This apparatus rotates the extraction vessels in an end-over-end fashion at 30.5 rpm.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). Millipore catalog number YT30 090 HW mechanical pressure Zero Head Space Extractor. This device is for use only when the waste is being tested for the mobility of volatile analytes (i.e., those identified as volatiles in ANALYTES section). The ZHE allows for liquid/solid separation within the device, and effectively precludes headspace. This vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see section 4.3.1). The vessels have an internal volume of 500mL, and are equipped to accommodate a 90mm filter. The devices contain VITON® O-rings which are replaced when visibly worn or when the piston within the ZHE can not be

moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted. The ZHE is checked for leaks one hour after initial pressurization. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

VITON® is a trademark of Du Pont.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is used. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Section 4.3.3). The filtration apparatus discussed in Section 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Apparatus: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in Section 4.2.1 is used for filtration.

[NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.]

4.3.2 Filter Apparatus: When the waste is evaluated for other than volatile analytes, a Millipore Corporation filter apparatus catalog number YT30142HW with a 100 psi maximum inlet, a 142mm filter diameter, and capable of holding 1.5 L is used. This devise uses positive pressure filtration.

4.3.3 Materials of Construction: Extraction vessels and filtration

devices are made of inert materials which will not leach or absorb waste components.

4.4 Filters: Environmental Express Acid Washed TCLP Filters. Catalog #FG77142MM. Filters are composed of binder free borosilicate glass fiber with a nominal particle retention of 0.7µ. Filters have been washed in 1N HNO3 solution and rinsed with three consecutive 1L aliquots of deionized water. Glass fiber filters are fragile and should be handled with care.

4.5 pH Meter: Orion bench top pH meter model 420A equipped with an Orion glass pH Sure-Flow™ electrode with Ag/AgCl internal reference system and an automatic temperature correction probe (ATC). The meter is accurate to +/- 0.05 units at 25°C.

4.6 ZHE Extract Collection Devices: TEDLAR® bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1% of total waste), the TEDLAR® bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

TEDLAR® is a registered trademark of Du Pont.

4.7 ZHE Extraction Fluid Transfer Device: Micropump® model #000-415 pump. This device transfers the extraction fluid into the ZHE without changing the nature of the extraction fluid.

4.8 Analytical balance- capable of accurate measurement to 0.01g.
(Sartorious PT 120 ID#30121328 (120g max))
(Mettler-Toledo PB602 (610g max))
Analytical balance- capable of accurate measurement to 0.0001g.
(Sartorious A200S)
(all weight measurements are to be within +/- 0.1 grams).

4.9 Beakers, 250 mL.

4.10 Watchglasses, appropriate diameter to cover 250 mL beaker.

4.11 Magnetic stir bars.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.(e.g.Mallinckrodt from VWR).

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an SMI TopSider Series liquid dispenser.

5.1.3 Nitric acid (conc), HNO3.(e.g.Tracepur Plus by EM from VWR).

5.1.4 Nitric acid (1:1), HNO3. Add 500 mL concentrated HNO3 to 400 mL water and dilute to 1 liter in an SMI Topsider Series 2 liquid dispenser.

5.1.5 Acetic acid, glacial. (e.g.Tracepur Plus by Em from VWR).

5.1.6 Extraction fluid:

5.1.6.1 Extraction fluid # 1: This is the most commonly used fluid and is, therefore, made 50 liters at a time in a carboy.
- add approximately 30L of DI h20 to carboy
- carefully add 285mL of acetic acid to carboy
- add 3215mLs of 1N NaOH to carboy
  -mix 127.85g NaOH pellets to 3215mLs DI and stir until dissolved
- bring to final volume of 50L and mix well
- when correctly prepared, the pH of this fluid will be 4.93 +/- 0.05.

5.1.6.2 Extraction fluid # 2: This fluid is not commonly used and is, therefore, made 20 liters at a time in a carboy.
- add approximately 15L of DI h_20 to carboy
- carefully add 114 mLs of acetic acid to carboy
- bring to final volume of 20L and mix well
- when correctly prepared, the pH of this fluid will be 2.88 +/- 0.05.

[Note: These extraction fluids are monitored each use to check for impurities. The pH is checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.]

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water must meet ASTM type II standards. Reagent water will be interference free. Reagent water is dispensed from a Barnstead Nano-Pure unit Model #D4741, Serial #687920145027. Cartridges are changed approximately every six months. The Barnstead unit is fed water from a Kinetico commercial reverse osmosis unit (serial #361001). This unit has the capability to produce 75 gallons of water per day and has a 20 gallon bladder tank for storage. This unit also has a 10", 5 micron prefilter which is changed every other time the cartridges are changed.

5.3 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles.

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations are acidified with 2.5 mL 1:1 nitric acid to pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. See table 3 for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations - Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Section 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Section 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter apparatus. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle.

7.1.1.7 Quantitatively transfer the waste sample to the filter apparatus (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over

what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

[Note: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.]

Gradually apply gentle air pressure of 1-10 psi, until air moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the air pressure has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the air pressure begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

[Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.]

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

[NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.]

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 7.1.1.9)}}{\text{Total weight of waste (section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in section 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to determine whether the solid material requires particle size reduction or to section 7.1.2.1 if it is

noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 7.1.1.9 is less than 0.5%, then proceed to section 7.2.9 if the nonvolatile TCLP is to be performed and to section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 +/- 20-C until two successive weighing yield the same value within +/- 1%. Record the final weight.

[NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.]

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial wt. of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to section 7.2.9 if the nonvolatile TCLP is to be performed, and to section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (section 7.1.3) and determine the appropriate extraction fluid (section 7.1.4). If only the volatile TCLP is to be performed, see the note in section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size.

Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Section 7.3.6).

[Note: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.]

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2), determine the appropriate fluid (Section 5.1.6) for the nonvolatiles extraction as follows:

[NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.1.6.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.]

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 250 mL beaker.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50°C, and hold at 50°C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation (Sections 7.1.1 - 7.1.4) was determined to be 100% solid at section 7.1.1.1, then it can be used for the section 7.2 extraction (assuming at least 100 grams remain), and the section 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 7.3. The aliquot of the waste subjected to the procedure in section 7.1.1.7 might be appropriate for use for the section 7.2 extraction if an adequate amount of solid (as determined by section 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to section 7.2.10 of the nonvolatile TCLP extraction.

7.2 Procedure When Volatiles are not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, see section 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of

concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see section 7.1.1), weigh out a subsample of the waste (100 gram minimum) and proceed to section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in section 4.3.2 and is outlined in sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter apparatus. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see section 4.4).

[NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.]

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (section 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (sections 7.1.1 or 7.1.2), use the percent solids information obtained in section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle.

7.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see section 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

[NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.2.5, to determine the weight of the waste sample that will be filtered.]

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

[NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.]

7.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (see section 7.2.12) or stored at 4°C until time of analysis.

[NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in section 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.]

7.2.9 If the waste contains <0.5% dry solids (see Section 7.1.2), proceed to Section 7.2.13. If the waste contains >0.5% dry solids (see section 7.1.1 or 7.1.2), and if particle size reduction of the solid was needed in section 7.].3, proceed to section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

[NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.]

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (section 7.1.1)} \times \text{weight of waste filtered (section 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see section 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 +/- 2 rpm for 18 +/- 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 +/- 2-C during the extraction period.

[NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.]

7.2.12 Following the 18 +/- 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see section 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from section 7.2.12 is defined as the TCLP extract. Proceed to section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from section 7.2.12 with the initial liquid phase of the waste obtained in section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from section 7.2.7, is not or may not be compatible with the filtered liquid resulting from section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion

of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to +/- 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V1)(C1) + (V2)(C2)}{V1 + V2}$$

where:

V1 = The volume of the first phase (L).
C1 = The concentration of the analyte of concern in the first phase (mg/L).
V2 - The volume of the second phase (L).
C2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (see section 4.6) and set aside. If using a TEDLAR bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in section 4.6 are recommended for use under the conditions stated in sections 4.6.1 - 4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it is helpful

to first moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2- usually about one inch from the top). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid (see section 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids (section 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing >= 0.5% dry solids (sections 7.1.1 and/or 7.1.2), use the percent solids information obtained in section 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For wastes containing < 0.5% solids (see section 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing >= 0.5% solids (see section 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent solids (Section 7.1.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in section 7.1.3, proceed to section 7.3.6. If particle size reduction was not required in section 7.1.3, proceed to section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in section 7.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4°C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

[NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative.

Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.]

When the surface area or particle size has been appropriately altered, proceed to section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom).

[NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in section 7.3.4 to determine the weight of the waste sample that will be filtered.]

Attach the compressed air line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

[NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.]

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

[NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.]

If the original waste contained <0.5% dry solids (see section 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (see sections 7.3.13 through 7.3.15) or stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (see section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid using the Micropump into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus and rotate at 30 +/- 2 rpm for 18 +/- 2 hours. Ambient temperature shall be maintained at 23 +/- 2°C during agitation.

7.3.13 Following the 18 +/- 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter,

using the ZHE device as discussed in section 7.3.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see sections 4.6 and 7.3.1).

[NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber

filter has been ruptured.]

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from section 7.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from section 7.3.13 and the initial liquid phase (section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4°C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V1)\ (C1) + (V2)\ (C2)}{V1 + V2}$$

where:

V1 = The volume of the first phases (L).
C1 = The concentration of the analyte of concern in the first phase (mg/L).
V2 = The volume of the second phase (L).
C2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to section 8.0 for quality assurance requirements.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed at a rate of one per 20 analytical samples.

8.2 A matrix spike need not be performed if the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. Follow the matrix spike addition guidance provided in the associated analytical method (see table 2 for inorganic analyte spiking concentrations).

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be

added prior to TCLP extraction of the sample.

8.2.2 In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R(\%Recovery) = 100(X(s) - X(u))/K$$

Where:

X(s) = measured value for the spiked sample,
X(u) = measured value for the unspiked sample, and
K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1 The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amount of standard to three of these aliquots. The fourth aliquot in unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentration are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentration

as the dependent variable(y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abcissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependent variable versus the independent variable. Derive concentrations for unknown using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo TCLP extraction within the time periods listed in table 3.

9.0 METHOD PERFORMANCE

9.1 Ruggedness. Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

9.1.1 Metals - The following conditions were used when leaching a waste for metals analysis:

Varying Conditions

Liquid/Solid ratio	19:1 vs. 21:1
Extraction time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	yes vs. no 1311- 20
Filter type	0.7 um glass fiber vs. 0.45 um vs. polycarbonate
Bottle type	borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within +/- 0.05 units as specified.

9.1.2 Volatile Organic Compounds - The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions

Liquid/Solid ratio	19:1 vs. 21:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar(R) bag
Aliquotting	yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

9.2 Precision. Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

9.2.1 Metals - The results of a multi-laboratory study are shown in Table 4, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification

requirements.

9.2.2 Semi-Volatile Organic Compounds - The results of two studies are shown in Tables 5 and 6. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual

compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical conditions used.

9.2.3 Volatile Organic Compounds - Eleven laboratories participated in a collaborative study of the use of the ZHE with two waste types which were fortified with a mixture of VOCs. The results of the collaborative study are shown in table 7. Precision results for VOCs tend to occur over a considerable range. However, the range and mean RSD compared very closely to the same collaborative study metals results in table 4. Blackburn and Show concluded that at the 95% level of significance: 1) recoveries among laboratories were statistically similar, 2) recoveries did not vary significantly between the two sample types, and 3) each laboratory showed the same pattern of recovery for each of the two samples.

10.0 REFERENCES

1. Blackburn, W.B. and Show, I. "Collaborative Study of the Toxicity Characteristics Leaching Procedure (TCLP)." Draft Final Report, Contract No. 6803-1958, S-Cubed, November 1986.
2. Newcomer, L.R., Blackburn, W.B., Kimmell, T.A. "Performance of the Toxicity Characteristic Leaching Procedure." Wilson Laboratories, S-Cubed, U.S. EPA, December 1986.
3. Williams, L.R., Francis, C.W.; Maskarinec, M.P., Taylor D.R., and Rothman, N. "Single-Laboratory Evaluation of Mobility Procedure for Solid Waste." EMSL, ORNL, S-Cubed, ENSECO.

Table 1. TCLP ANALYSIS AND TOTALS ANALYSIS RELATION

TCLP analyses are not a reflection of the total amount present in a sample and should never be assumed as total contaminant concentrations. Likewise samples that have no detectable limits in a TCLP analysis should never be assumed to be below detectable limits for total contaminant analyses. Total concentrations, however, do have a relationship with TCLP concentrations. The regulation for TCLP in 40CFR part 261 states that if a total contaminant concentration shows that the regulated TCLP limits for the contaminant will not be exceeded even if all of the contaminant leaches out of the sample, a TCLP analyses need not be performed. The factor used to calculate this relationship is 20. This means that if by taking a total concentration expressed in mg/kg and dividing it by 20, the resulting number is less than the regulated limit (in mg/L), a TCLP analysis for that contaminant is not necessary. The factor of 20 is determined as follows:

0.100 kg sample/2.00 L extraction fluid = 0.05 = 1/20

therefore- for a total lead concentration of 75 mg/kg

75 mg/kg * 0.100 kg = 7.5 mg lead
7.5 mg lead/ 2.00 L of leachate = 3.8 mg/L TCLP

even if all of the lead in the sample leached out the maximum TCLP value could only reach 3.8 mg/L. The regulated limit is 5.0 mg/L and could not be exceeded. By the TCLP regulation, this sample does not have to be tested for leachability.

The bottom line is that a total analysis is a measurement of the total contaminant concentration in the sample matrix. The TCLP analysis shows only the leachable amount of the contaminant present. The results of this analysis are variable and differ greatly depending on pH, matrix type, surface area and solubility. In any case, the TCLP results will never be the same numeric value as the total results and TCLP analysis should never be substituted for total contaminant analysis.

TABLE 2. SPIKING CONCENTRATIONS

Element	Stock concentrations (ug/mL)	Spike amt. water matrix (ug/L)
As	2000	4000
Ba	2000	4000
Cd	50	100
Cr	200	400
Pb	500	1000
Se	2000	4000
Ag	50	100

these concentrations are obtained by diluting 200 uL for water matrix with 100mL final volume
each of the three-spike-set from Inorganic Ventures, Inc. Cat# CLPP-SPK-SET.

Table 3 SAMPLE MAXIMUM HOLDING TIMES (days)

	From: Field Collection To: TCLP extraction	From: TCLP extraction To:Preparative extraction	From: Preparative extraction To: determinative analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semivolatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

Table 4. MULTI-LABORATORY TCLP METALS, PRECISION

Waste	Extraction Fluid	Metal	$\bar{X}$	S	%RSD
Ammonia	#1	Cadmium	0.053	0.031	60
Lime Still	#2		0.023	0.017	76
Bottoms	#1	Chromium	0.015	0.0014	93
	#2		0.0032	0.0037	118
	#1	Lead	0.0030	0.0027	90
	#2		0.0032	0.0028	87
API/EW	#1	Cadmium	0.0046	0.0028	61
Mixture	#2		0.0005	0.0004	77
	#1	Chromium	0.0561	0.0227	40
	#2		0.105	0.018	17
	#1	Lead	0.0031	0.0031	100
	#2		0.0124	0.0136	110

Fossil	#1	Cadmium	0.080	0.069	86
Fuel Fly	#2		0.093	0.067	72
Ash	#1	Chromium	0.017	0.014	85
	#2		0.070	0.040	57
	#1	Lead	0.0087	0.0074	85
	#2		0.0457	0.0083	18

%RSD Range = 17 - 118
Mean %RSD = 74
Note: X = Mean results from 6 - 12 different laboratories
Units = mg/L
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 5. SINGLE-LABORATORY SEMI-VOLATILES, PRECISION

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	Phenol	#1	19000	2230	11.6
		#2	19400	929	4.8
	2-Methylphenol	#1	2000	297	14.9
		#2	1860	52.9	2.8
	4-Methylphenol	#1	7940	1380	17.4
		#2	7490	200	2.7
	2,4-Dimethylphenol	#1	321	46.8	14.6
		#2	307	45.8	14.9
	Naphthalene	#1	3920	413	10.5
		#2	3827	176	4.6
	2-Methylnaphthalene	#1	290	44.8	15.5
		#2	273	19.3	7.1
	Dibenzofuran	#1	187	22.7	12.1
		#2	187	7.2	3.9
	Acenaphthylene	#1	703	89.2	12.7
		#2	663	20.1	3.0
	Fluorene	#1	151	17.6	11.7
		#2	156	2.1	1.3
	Phenanthrene	#1	241	22.7	9.4
		#2	243	7.9	3.3
	Anthracene	#1	33.2	6.19	18.6
		#2	34.6	1.55	4.5
	Fluoranthrene	#1	25.3	1.8	7.1
		#2	26.0	1.8	7.1
API/EW Mixture	Phenol	#1	40.7	13.5	33.0
		#2	19.0	1.76	9.3
	2,4-Dimethylphenol	#1	33.0	9.35	28.3
		#2	43.3	8.61	19.9
	Naphthalene	#1	185	29.4	15.8
		#2	165	24.8	15.0
	2-Methylnaphthalene	#1	265	61.2	23.1
		#2	200	18.9	9.5

%RSD Range = 1 - 33
Mean %RSD = 12
Note: Units = ug/L
Extractions were performed in triplicate
All results were at least 2x the detection limit
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 6. MULTI-LABORATORY SEMI-VOLATILES, PRECISION

Waste	Compound	Extraction Fluid	X	S	%RSD
Ammonia Lime Still Bottoms (A)	BNAs	#1	10043	7680	76.5
		#2	10376	6552	63.1
API/EW Mixture (B)	BNAs	#1	1624	675	41.6
		#2	2074	1463	70.5
Fossil Fuel Fly Ash (C)	BNAs	#1	750	175	23.4
		#2	739	342	46.3

Mean %RSD = 54

Note: Units = ug/L
X = Mean results from 3 - 10 labs
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

%RSD Range for Individual Compounds

A, #1	0 - 113
A, #2	28 - 108
B, #1	20 - 156
B, #2	49 - 128
C, #1	36 - 143
C, #2	61 - 164

Table 7. MULTI-LABORATORY (11 LABS) VOCS, PRECISION

Waste	Compound	$\bar{X}$	S	%RSD
Mine Tailings	Vinyl chloride	6.36	6.36	100
	Methylene chloride	12.1	11.8	98
	Carbon disulfide	5.57	2.83	51
	1,1-Dichloroethene	21.9	27.7	127
	1,1-Dichloroethane	31.4	25.4	81
	Chloroform	46.6	29.2	63
	1,2-Dichloroethane	47.8	33.6	70
	2-Butanone	43.5	36.9	85
	1,1,1-Trichloroethane	20.9	20.9	100
	Carbon tetrachloride	12.0	8.2	68
	Trichloroethene	24.7	21.2	86
	1,1,2-Trichloroethene	19.6	10.9	56
	Benzene	37.9	28.7	76
	1,1,2,2-Tetrachloroethane	34.9	25.6	73
	Toluene	29.3	11.2	38
	Chlorobenzene	35.6	19.3	54
	Ethylbenzene	4.27	2.80	66
	Trichlorofluoromethane	3.82	4.40	115
	Acrylonitrile	76.7	110.8	144
Ammonia Lime Still Bottoms	Vinyl chloride	5.00	4.71	94
	Methylene chloride	14.3	13.1	92
	Carbon disulfide	3.37	2.07	61
	1,1-Dichloroethene	52.1	38.8	75
	1,1-Dichloroethane	52.8	25.6	49
	Chloroform	64.7	28.4	44
	1,2-Dichloroethane	43.1	31.5	73
	2-Butanone	59.0	39.6	67
	1,1,1-Trichloroethane	53.6	40.9	76
	Carbon tetrachloride	7.10	6.1	86
	Trichloroethene	57.3	34.2	60
	1,1,2-Trichloroethene	6.7	4.7	70
	Benzene	61.3	26.8	44
	1,1,2,2-Tetrachloroethane	3.16	2.1	66
	Toluene	69.0	18.5	27
	Chlorobenzene	71.8	12.0	17
	Ethylbenzene	3.70	2.2	58
	Trichlorofluoromethane	4.05	4.8	119
	Acrylonitrile	29.4	34.8	118

%RSD Range = 17 - 144
Mean %RSD = 75
Note: Units = ug/L

Attachment QAPP-B8

Acid Digestion of Aqueous Samples & Extracts for Total Metals
Method 3010A

GAM 3010A
ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY FLAA OR ICP SPECTROSCOPY
Revision 3.01: 02/08/02

ANALYTES:

		CAS #
Aluminum	(Al)	7429-90-5
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-43-9
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Sodium	(Na)	7440-23-5
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

1.0 SCOPE AND APPLICATION

1.1 This digestion procedure is used for the preparation of aqueous samples, TCLP extracts, and wastes that contain suspended solids for analysis, by flame atomic absorption spectroscopy (FLAA) or inductively coupled argon plasma spectroscopy (ICP-AES) or inductively coupled argon mass spectrometry. The procedure is used to determine total metals.

2.0 SUMMARY OF METHOD

2.1 A mixture of nitric acid and the material to be analyzed is refluxed in a covered Environmental Express digestion cup. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If sample should go to dryness, it must be discarded and the sample re-prepared.

3.0 INTERFERENCES

3.1 The analyst should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy some metal complexes.

3.2 Precipitation will cause a lowering of the silver concentration and therefore an inaccurate measurement. For the acid combination used in this method, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.

4.0 APPARATUS AND MATERIALS

4.1 Class A volumetric flasks: 5-, 10-, 25-, 50-, and 100-mL

4.2 Varian Vista-MPX ICP-OES, and UltraMass ICP-MS

4.3 Environmental Express(E.E.) Hot Block Digestion Unit

4.4 E.E.ribbed watch glasses

4.5 E.E.70ml digestion vessels with 5ml increments

4.6 E.E. filter and plunger units

4.7 MLA Precision air displacement pipetters: 10, 25, 50, 100, 250, 1000µL single volume. MLA variable volume 10-50µl and 200-1000µl.

4.8 Analytical balance- capable of accurate measurement to 0.01g.
(Sartorious PT 120 ID#30121328 (120g max))
(Mettler-Toledo PB602 (610g max))
Analytical balance- capable of accurate measurement to 0.0001g.
(Sartorious A200S)

4.9 Boekel oven (model#107801)

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.(Omnitrace EM Science cat.no. HX0607-2 from VWR).

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an SMI TopSider Series 2 liquid dispenser.

5.1.3 Nitric acid (conc), HNO_3.(Omnitrace EM Science cat.no. NX0407-2 from VWR).

5.1.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an SMI Topsider Series 2 liquid dispenser.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water must meet ASTM type II standards. Reagent water will be interference free. Reagent water is dispensed from a Barnstead Nano-Pure unit Model #D4741, Serial #687920145027. Cartridges are changed approximately every six months. The Barnstead unit is fed water from a Kinetico commercial reverse osmosis unit (serial #361001). This unit has the capability to produce 75 gallons of water per day and has a 20 gallon bladder tank for storage. This unit also has a 10", 5 micron prefilter which is changed every other time the cartridges are changed.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous samples shall be preserved to pH <2 with nitric acid and have a holding time of 6 months for analysis of all metals except mercury which has a holding time of 28 days. Soil samples have a holding time of 6 months for all metals except mercury which has a holding time of 28 days.

7.0 PROCEDURE

7.1 Turn on the hot block and allow it to reach 90-95°C. This will take approximately 20-30 minutes. Use a digestion cup w/a thermometer to monitor the temperature of the hot block.

7.2 Using an E.E. digestion vessel, measure 50 mL of the extract. For TCLP samples, measure out a 5 ml aliquot and dilute the extract to 50 mL with deionized water. The vessel(s) must be labeled with the correct sample number using a permanent marker. All matrix spikes, duplicates, control samples and blanks should be prepared by following the same procedures as the samples.

7.3 To the sample dispense 1.5 mL of concentrated HNO_3 from the individually labeled fixed volume repipet dispensers located in the digestion hood.

7.4 Cover the beaker with a ribbed watch glass and carefully place the beaker onto the hot block, at a temperature of 95°C.

7.5 The sample will remain on the hot block as long as it takes to reduce the volume to about 5 mL. Care must be taken not to allow the sample to boil as analyte loss may occur. Sample must not be allowed to go to dryness. If either of these things happen, the sample must be redigested using the steps above.

7.6 After volume is reduced, allow sample to cool in the hood. Then add another 1.5ml portion of concentrated HNO3, and place on the hot block.

7.7 Continue heating (~1-2 hrs) to a low volume.

7.8 Remove the vessel(s)and allow to cool.

7.9 Add 5mls of 1:1 HCl. Cover the beaker with a solid watch glass, and let reflux for 15-30 minutes to dissolve any precipitate of residue resulting from evaporation.

7.10 Remove sample(s) from hot block and allow to cool.

7.11 Rinse the watch glass into the sample using deionized water.

7.12 Bring the sample to final volume of 50 mL and gently shake. Sample is now ready for analysis.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples, whichever is more frequent. Associated methods will dictate spiking concentrations.

8.2 CORRECTIVE ACTIONS

8.2.1 Samples associated with control samples and blanks that fall outside of the acceptance criteria of the associated method must be re-digested.

8.2.2 Samples associated with matrix spikes and duplicates that fall outside of the acceptance criteria of the associated method must be flagged as "estimated concentration."

8.2.3 Samples must be re-digested if they boil or are evaporated to dryness.

Attachment QAPP-B9

Acid Digestion of Water for Total or Dissolved Metals
Method 3005A

GAM 3005A
ACID DIGESTION OF WATER FOR TOTAL RECOVERABLE OR DISSOLVED METALS FOR ANALYSIS BY FLAA OR ICP SPECTROSCOPY
Revision 5.0: 2/08/2002

ANALYTES:

		CAS #
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-43-9
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare surface and ground water samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP-AES) or inductively coupled plasma mass spectroscopy (ICP-MS).

1.2 When analyzing for total dissolved metals the sample is filtered at the time of collection, prior to acidification with nitric acid.

2.0 SUMMARY OF METHOD

2.1 Total recoverable metals-The entire sample is acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and substatially reduced in volume. The digestate is filtered and dilluted to volume, and is then ready for analysis.

2.2 Dissolved metals-The sample is filtered through a 0.45µ filter at the time of collection and the liquid phase is then acidified with nitric acid. Samples for dissolved metals do not need to be digested as long as the acid concentrations have have been adjusted to the same concentrattion as the standards.

3.0 INTERFERENCES

3.1 The analyst should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy some metal complexes.

3.2 Precipitation will cause a lowering of the silver concentration and therefore an inaccurate measurement. For the acid combination used in this method, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stabele in a tap-water matrix for 30 days. Higher concentrations of silver requre additional HCL.

4.0 APPARATUS AND MATERIALS

4.1 Class A volumetric flasks: 5-, 10-, 25-, 50-, and 100-mL.

4.2 Varian Vista-MPX ICP-OES, and Ultramass ICP-MS

4.3 Environmental Express (E.E.) Hot Block digestion unit.

4.4 Environmental Express ribbed watch glasses.

4.5 Environmental Expess 70mL digestion vessels w/5mL increment markers.

4.6 Environmental Express filter and plunger units.

4.7 MLA Precision air displacement pipetters: 10, 25, 50, 100, 250, 1000µL single volume. MLA variable volume 10-50µl and 200-1000µl.

4.8 Analytical balance- capable of accurate measurement to 0.01g.
(Mettler-Toledo PB602 (610g max))
Analytical balance- capable of accurate measurement to 0.0001g.
(Sartorious A200S)

4.9 Boekel oven model 107801.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.(Omnitrace EM Science cat.no.HXO607-2 from VWR).

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mLs water and dilute to 1 liter in a repipet II liquid dispenser.

5.1.3 Nitric acid (conc), HNO_3. (Omnitrace EM Science cat.no NXO407-2 from VWR).

5.1.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mLs water and dilute to 1 liter in a repipet II liquid dispenser.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water must meet ASTM type II standards. Reagent water will be interference free. Reagent water is dispensed from a Barnstead Nano-Pure unit Model #D4741, Serial #687920145027. Cartridges are changed approximately every six months. The Barnstead unit is fed water from a Kinetico commercial reverse osmosis unit (serial #361001). This unit has the capability to produce 75 gallons of water per day and has a 20 gallon bladder tank for storage. This unit also has a 2 10", 5 micron prefilter which is changed every three months.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous samples shall be preserved to pH <2 with nitric acid and have a holding time of 6 months for analysis of all metals except mercury which has a holding time of 28 days. Soil samples have a holding time of 6 months for all metals except mercury which has a holding time of 28 days.

6.2 Total recoverable metals-All samples must be acidified at the time of collection with 1:1 HNO_3 (approximatelly 5-10 mL/L or more to achieve a pH<2).

6.3 Dissolved metals -All samples must be filtered through a 0.45 µm filter and then acidified at the time of collection with 1:1 HNO_3 (approximatelly 5-10 mL/L or more to achieve a pH<2).

7.0 PROCEDURE

7.1 Turn on hot block and allow it to reach 90-95°C. This will take approximately 20-30 minutes.

7.2 Using an Environmental Express digestion vessel measure out 50 mLs of the water sample and pour into the 70 mL graduated digestion vessel. The beaker must be labeled with the correct sample number using a permanent marker. All matrix spikes, duplicates, control samples and

blanks should be prepared by following the same procedures as the samples.

7.3 To the sample dispense 1 mL of concentrated HNO_3 and 2.5 mls conc. HCl from the individually labeled fixed volume repipet dispensers located in the digestion hood.

7.4 Cover the vessels with a ribbed watch glass and carefully place them into the preheated block digestion unit.

7.5 The sample will remain on the hot block as long as it takes to reduce the volume to 15-20 mLs. Care must be taken to not allow the sample to boil as analyte loss may occur. Sample must not be allowed to go to dryness. If either of these things happen sample must be redigested using the steps above.

7.6 After volume is reduced, allow sample to cool in the hood.

7.7 Rinse the ribbed watch glass into the sample using deionized water from a squeeze bottle taking care to use a minimal amount so the final volume of 50 mL is not exceeded.

7.8 Bring the sample to final volume of 50 mL and shake. Sample is now ready for analysis.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent. Associated methods will dictate spiking concentrations.

9.0 CORRECTIVE ACTIONS

9.1.1 Samples associated with control samples and blanks that fall outside of the acceptance criteria of the associated method must be redigested.

9.1.2 Samples associated with matrix spikes and duplicates that fall outside of the acceptance criteria of the associated method must be flagged as "estimated concentration."

9.1.3 Samples must be re-digested if they boil or are evaporated to dryness.

Attachment QAPP-B10

Ultrasonic Extraction of Solid Matrices
Method 3550A

GAM 3550A
Ultrasonic Extraction of Solid Matrices
Rev. 3.0 2/1/02

1.0 SCOPE AND APPLICATION

1.1 Method 3550 is a procedure for, extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. The ultrasonic process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 The method is divided into two sections, based on the expected concentration of organics in the sample. The low concentration method (individual organic components of (< 20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The medium/high concentration method (individual organic components of > 20 mg/kg) is much simpler and therefore faster.

1.3 It is highly recommended that the extracts be cleaned up prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Low concentration method - A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

2.2 Medium/high concentration method - A 2 g sample is mixed with anhydrous sodium sulfate. This mixture is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by filtration. The extract is ready for cleanup and/or analysis following concentration.

3.0 INTERFERENCES

3.1 Method specific see specific analytical method.

3.2 All glassware must be cleaned according to organic specifications and pre-rinsed with the extraction solvent prior to proceeding with the extraction to minimize organic interferences.

3.3 Phthalate esters are commonly used as plasticizers and present a potential source of contamination. All plastic materials, other than Teflon, should be avoided in the preparation process.

3.5 Some interferences may be present in the samples and may be minimized or removed using the appropriate cleanup procedure.

4.0 APPARATUS AND MATERIALS

4.2 Ultrasonic preparation - Solid 3/4 in horn

4.2.1 Ultrasonic Disrupter - Tekmar model TM375, 375 Watts power rating. Tuned using the manufacturers instructions for preparing the disrupter for extraction of samples with low and medium/high concentration. Use a 3/4" horn for the low concentration method and a 1/8" tapered microtip attached to a 1/2" horn for the medium/high concentration method.

4.2.2 Ultrasonic Disrupter - Virtis model Virsonic300, 400 Watts power rating. Tuned using the manufacturers instructions for preparing the disrupter for extraction of samples with low and medium/high concentration.

Use a 3/4" horn for the low concentration method and a 1/8" tapered microtip attached to a 1/2" horn for the medium/high concentration method.

4.3 Sonaboxes - Used with both above disrupters for decreasing noise.

4.4 Apparatus for determining percent dry weight.

4.4.1 Oven - Drying.
4.4.2 Desiccator.
4.4.3 Weighing pans -disposable aluminum.

4.5 Pasteur glass pipettes - 1 mL, disposable.

4.6 Beakers - 250 mL.

4.7 Vacuum filtration apparatus.

4.7.1 Vacuum Filtration apparatus, Kontes #953825-0000 or equivalent.
4.7.2 Filter paper - Gelman Nylaflo Nylon 0.45 µm or equivalent.

4.8 Zymark Turbovap, six position nitrogen evaporator.

4.9 Turbovap concentrator tubes 300 mL with 1 mL reservoir.

4.10 Turbovap tube racks six position.

4.11 Balance - Sartorious P120 Top loading, capable of accurately weighing to the nearest 0.01 g.

4.12 Vials - 2 mL, for GC autosampler, with Teflon lined screw caps or crimp tops.

4.13 Glass scintillation vials - 20 mL, with Teflon or aluminum foil lined screw caps.

4.14 Spatula - Stainless steel or Teflon.

4.15 Drying column - 300 mm x 20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom and Teflon stopcock. Use a small pad of Pyrex glass wool to retain the adsorbent. **Prerinse packed column with 50 mL of solvent used in extraction.**

4.16 Syringe - 1.0 mL.

4.17 Kuderna-Danish (K-D) apparatus.

4.17.1 Concentrator tube - 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.

4.17.2 Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

4.17.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

4.17.4 Snyder column - Two ball micro (Kontes K-569001-0219 or equivalent).

4.17.5 Plastic clamps - 1/2 inch (Kontes K-662750 or equivalent).

4.18 Boiling chips - Solvent extracted, approximately 10/40 mesh Teflon

4.19 Water bath - Heated, with concentric ring cover, capable of temperature control (+/- 5 C). The batch should be used in a hood.

5.0 REAGENTS

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise specified, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

5.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water. For semivolatiles and nonvolatile, all references to water in the methods refer to water in which an interferent is not observed at the method detection limit of the

compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

5.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400-C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

5.4 Extraction solvents.

5.4.1 Low concentration soil/sediment and aqueous sludge samples shall be extracted using a solvent system that gives optimum, reproducible recovery for the matrix/analyte combination to be measured. Suitable solvent choices are given in Table 1.

5.4.2 Methylene chloride : Acetone, CH_2Cl_2:CH_3COCH_3 (1:1, v:v). Pesticide quality or equivalent.

5.4.3 Methylene chloride, CH_2Cl_2. Pesticide quality or equivalent.

5.4.4 Hexane, C_6H_{14}. Pesticide quality or equivalent.

5.4.4 Hexane : Acetone, C_6H_{14}: CH_3COCH_3 (1:1, v:v). Pesticide quality or equivalent.

5.4.5 Freon 113, fluorocarbon-113 (1,1,2-trichloro-1,2,2-trifluoroethane): Mid-Atlantic Chemical.

5.5 Exchange solvents.

5.5.1 Hexane, C_6H_{14}. Pesticide quality or equivalent.
5.5.2 Cyclohexane, C_6H_{12}. Pesticide quality or equivalent.

5.7 Surrogate and Matrix Spiking Solutions (see Table 3 for appropriate volumes added for each method.)

5.7.1 8015 DRO Spiking Mix - DRO mix 2,000ug/mL Restek cat. no. 31064.

5.7.2 418.1 - Spike solution (Accustandard cat. no. M-418)neat components in the following parts by volume: Chlorobenzene 10.0,

n-Hexadecane 15.0, Isooctane 15.0.

5.7.3 8081 Pesticide Matrix Spike Mix, Restek cat. no. 32018. 25-50 ug/ml.

5.7.4 8081 Pesticide Surrogate Mix, Restek cat. no. 32000. 200 ug/ml.

5.7.5 8100 Spike Mix, Restek cat. no. 31011 diluted to 200 ug/ml in acetone.

5.7.6 8100 Surrogate Mix - 2-Fluorobiphenyl 99%(Aldrich catalog #10,274-1)and ortho-Terphenyl 99% (cat# T280-0)to a concentration of 20 ug/mL.

5.7.7 8121 Spike Mix: AccuStandard cat# 8121M Diluted to 0.05/0.50/5.0 (same concentration as initial calibration point #3 from table 4, GAM 8121.

5.7.8 1000 mg/L of 1,4-dichloronaphthalene and dilute it to 10 µg/uL.

5.7.9 8270 Matrix Spike Mix - Acid Compounds at 150 µg/mL and Base/Neutral Compounds at 100 µg/mL.

5.7.10 8270 Surrogate standards - Acid surrogates (RESTEK #31087) phenol-d(6), 2-fluorophenol, 2,4,6-tribromophenol at 150 µg/mL. Base Neutral surrogates (RESTEK #31086) nitrobenzene-d(5), 2-fluorobiphenyl, and p-terphenyl-d(14) at 100 µg/mL.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Method specific volumes and preservation techniques. See specific methods for the sample collection guidelines.

6.2 Soil samples are collected in 125 mL or 250 mL wide mouth precleaned glass containers with Teflon lined lids. Refrigerated to 4°C from time of collection to sample analysis.

7.0 PROCEDURE

7.1 Sample handling

7.1.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

7.1.1.1 Determine the dry weight of the sample (Section 7.2) remaining after decanting, if required.

7.1.2 Waste samples - Samples consisting of multiphases must be prepared by the phase separation method before extraction. This procedure is for solids only.

7.1.3 Dry waste samples amenable to grinding - Grind or otherwise subdivide the waste so that it either passes through a 1 mm sieve or can be extruded through a 1 mm hole. Introduce sufficient sample into the grinder to yield at least 100 g after grinding.

7.1.4 Gummy, fibrous or oily materials not amenable to grinding should

be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling of these difficult matrices.

7.2 Determination of percent dry weight - In certain cases, sample results are desired based on a dry weight basis. When such data is desired, or required, a portion of sample for this determination should be weighed out at the same time as the portion used for analytical determination. (WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated hazardous waste sample.) However, samples known or suspected to contain significant concentrations of toxic, flammable, or explosive constituents should not be over dried because of concerns for personal safety.

*Discretion is advised. It may be prudent to delay over drying of the weighed-out portion until other analytical results are available.

7.2.1 Immediately after weighing the sample for extraction, weigh 5 g of the sample into a tared aluminum pan. Determine the % dry weight of the sample by drying overnight at 105-C. Allow to cool in a desiccator before weighing:

$$\% \text{ dry weight} = \frac{\text{grams of dried sample}}{\text{grams of sample}} \times 100$$

7.3 Extraction method for samples expected to contain low concentrations of organics and pesticides (< 20 mg/kg):

7.3.1 The following step should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample into a 250 mL beaker. Record the weigh to the nearest 0.1 g. Nonporous or wet samples (gummy or clay type) that do not have a free-flowing sandy texture must be mixed with 60 g of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate may be added. After addition of sodium sulfate, the sample should be free flowing. Add 1 mL of surrogate standards to all samples, spikes, standards, and blanks. Refer to Table 3 for each determinative method to be used, for details on the surrogate standard solution and the matrix spike solution). For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 10 µg/mL of each base/neutral analyte and 15 µg/mL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). Add approximately 100 mL of the solvent selected from table 1.

7.3.1.1 For each analytical batch or 20 samples, a preparation blank, laboratory control sample (LCS), matrix spike (MS) and matrix spike duplicate must be processed.

7.3.1.2 Refer to table 3 for the appropriate volume of Spiking solution for each specific semivolatiles method being used.

7.3.2 Place the bottom surface of the tip of the 3/4 in. disrupter horn about 1/2 in. below the surface of the solvent, but above the sediment layer.

7.3.3 Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% (energy on 50% of time and off 50%. of time). Do not use microtip probe.

7.3.4 Decant and filter extracts through a glass column (4.15)with 10 cm of sodium sulfate on top of a pre-rinsed glass wool pad. The column is pre-wet with 20 mL of $MeCl_2$ and stopped before the top of the sodium sulfate is reached.

7.3.5 Repeat the extraction two or more times with two additional 100 ml portions of solvent. Decant off the solvent after each ultrasonic extraction and pass through the column in 7.3.4. Collect and combine all three extracts in a Zymark Turbovap concentrator tube. Extracts must be stored at 4° C until analysis. Proceed to step 7.3.6.

7.3.6 Nitrogen Blowdown Technique

7.3.6.1 Place the concentrator tube in a warm water bath (approximately 35-C) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

7.3.6.2 The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry. Extracts must be stored at 4° C until analysis.

CAUTION: When the volume of solvent is reduced below 0.5 mL, semivolatile analytes may be lost.

7.3.7 Kuderna Danish Concentration Method

7.3.7.1 Assemble a Kuderna-Danish (K-D) concentrator (if necessary) by attaching a 10 mL concentrator tube to a 500 mL evaporator flask. Transfer filtered extract to a 500 mL evaporator flask and proceed to the next section.

7.3.7.2 Add one to two clean boiling chips to the evaporation flask, and attach a three ball Snyder column. Prewet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90-C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood with condensed solvent.

When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

7.3.7.3 If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add 50 mL of the exchange solvent and a new boiling chip, and re-attach the Snyder column. Concentrate the extract as described in Section 7.3.7, raising the temperature of the water bath, if necessary, to maintain proper distillation. When the apparent volume again reaches 1-2 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

7.3.7.4 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to GAM 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Section 7.3.6 or adjusted to 1 to 10.0 mL with the solvent last used.

7.3.7.5 If further concentration is indicated in Table 2, either micro Snyder column technique (Section 7.3.7.6) or nitrogen blow down technique (Section 7.3.6) is used to adjust the extract to the final volume required. Extracts must be stored at 4° C until analysis.

7.3.7.6 Micro Snyder Column Technique

7.3.7.6.1 Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding approximately 0.5 mL of methylene chloride or exchange solvent through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature, as required, to complete the concentration in 5-10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the liquid reaches an apparent volume of approximately 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its lower joint with approximately 0.2 mL of appropriate solvent and add to the concentrator tube. Adjust the final volume to the volume required for cleanup or for the determinative method (see Table 2).

7.4 If analysis of the extract will not be performed immediately, cover the concentrator tube and store refrigerated. If the extract will be stored longer than 2 days, it should be transferred to a vial with a

Teflon lined cap and labeled appropriately. Extracts must be stored at 4° C until analysis.

7.5 Extraction method for samples expected to contain high concentrations of organics (> 20 mg/kg):

7.5.1 Transfer approximately 2 g (record weight to the nearest 0.1 g) of sample to a 20 mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross contamination.

7.5.2 Add 2 g of anhydrous sodium sulfate to sample in the 20 mL vial and mix well.

7.5.3 Surrogate standards are added to all samples, spikes, and blanks (see Table 3 for details on the surrogate standard solution and

on the matrix spike solution). Add 2.0 mL of surrogate spiking solution to sample mixture. For the sample in each analytical batch selected for spiking, add 2.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 200 ng/uL of each base/neutral analyte and 300 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection).

7.5.4 Immediately add whatever volume of solvent is necessary to bring the final volume to 10.0 mL considering the added volume of surrogates and matrix spikes. Disrupt the sample with the 1/8 in. tapered microtip ultrasonic probe for 2 minutes at output control setting 5 and with mode switch on pulse and percent duty cycle at 50%. Extraction solvents are:

1. Nonpolar compounds (i.e., organochlorine pesticides and PCBs), hexane or appropriate solvent.
2. Extractable priority pollutants, methylene chloride.

7.5.5 Loosely pack disposable Pasteur pipettes with 2 to 3 cm Pyrex glass wool plugs. Filter the extract through the glass wool and collect 5.0 mL in a concentrator tube if further concentration is required. Follow Section 7.3.11 for details on concentration. Normally, the 5.0 mL extract is concentrated to approximately 1.0 ml or less.

7.5.6 The extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives.

8.0 QUALITY CONTROL

8.1 Any reagent blanks, matrix spike and laboratory control samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to the specific method for specific quality control procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative method for performance data.

10.0 REFERENCES

10.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

10.2 U.S. EPA, Interlaboratory Comparison Study: Methods for Volatile and Semi-Volatile Compounds, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, NV, EPA 600/4-84-027, 1984.

10.3 Christopher S. Hein, Paul J. Marsden, Arthur S. Shurtleff, "Evaluation of Methods 3540 (Soxhlet) and 3550 (Sonication) for Evaluation of Appendix IX Analytes form Solid Samples", S-CUBED, Report for EPA Contract 68-03-33-75, Work Assignment No. 03, Document No. SSS-R-88-9436, October 1988.

TABLE 1
EFFICIENCY OF EXTRACTION SOLVENT SYSTEMS(a)

			Solvent System (a)									
			A		B		C		D		E	
ompound	CAS No(b)	ABN(c)	%R	SD	%R	SD	%R	SD	%R	SD	%R	SD
-Bromophenyl phenyl ether	101-55-3	N	64.2	6.5	56.4	0.5	86.7	1.9	84.5	0.4	73.4	1.0
-Chloro-3-methylphenol	59-50-7	A	66.7	6.4	74.3	2.8	97.4	3.4	89.4	3.8	84.1	1.6
is(2-Chloroethoxy)methane	111-91-1	N	71.2	4.5	58.3	5.4	69.3	2.4	74.8	4.3	37.5	5.8
is(2-Chloroethyl) ether	111-44-4	N	42.0	4.8	17.2	3.1	41.2	8.4	61.3	11.7	4.8	1.0
-Chloronaphthalene	91-58-7	N	86.4	8.8	78.9	3.2	100.8	3.2	83.0	4.6	57.0	2.2
-Chlorophenyl phenyl ether	7005-72-3	N	68.2	8.1	63.0	2.5	96.6	2.5	80.7	1.0	67.8	1.0
2-Dichlorobenzene	95-50-1	N	33.3	4.5	15.8	2.0	27.8	6.5	53.2	10.1	2.0	1.2
3-Dichlorobenzene	541-73-1	N	29.3	4.8	12.7	1.7	20.5	6.2	46.8	10.5	0.6	0.6
ethyl phthalate	84-66-2	N	24.8	1.6	23.3	0.3	121.1	3.3	99.0	4.5	94.8	2.9
6-Dinitro-o-cresol	534-52-1	A	66.1	8.0	63.8	2.5	74.2	3.5	55.2	5.6	63.4	2.0
4-Dinitrotoluene	121-14-2	N	68.9	1.6	65.6	4.9	85.6	1.7	68.4	3.0	64.9	2.3
6-Dinitrotoluene	606-20-2	N	70.0	7.6	68.3	0.7	88.3	4.0	65.2	2.0	59.8	0.8
eptachlor epoxide	1024-57-3	N	65.5	7.8	58.7	1.0	86.7	1.0	84.8	2.5	77.0	0.7
exachlorobenzene	118-74-1	N	62.1	8.8	56.5	1.2	95.8	2.5	89.3	1.2	78.1	4.4
exachlorobutadiene	87-68-3	N	55.8	8.3	41.0	2.7	63.4	4.1	76.9	8.4	12.5	4.6
exachlorocyclopentadiene	77-47-4	N	26.8	3.3	19.3	1.8	35.5	6.5	46.6	4.7	9.2	1.7
exachloroethane	67-72-1	N	28.4	3.8	15.5	1.6	31.1	7.4	57.9	10.4	1.4	1.2
-Nitro-o-toluidine	99-55-8	B	52.6	26.7	64.6	4.7	74.7	4.7	27.9	4.0	34.0	4.0
itrobenzene	98-95-3	N	59.8	7.0	38.7	5.5	46.9	6.3	60.6	6.3	13.6	3.2
enol	108-95-2	A	51.6	2.4	52.0	3.3	65.6	3.4	65.5	2.1	50.0	8.1
2,4-Trichlorobenzene	120-82-1	N	66.7	5.5	49.9	4.0	65.6	3.4	65.5	2.1	50.0	8.1

(a) Percent recovery of analytes spiked at 200 mg/KG into NIST sediment SRM 1645
b) Chemical Abstracts Service Registry Number
c) Compound Type: A= Acid, B = Base, N = Neutral
d) A = Methylene chloride
B = Methylene chloride/Acetone (1/1)
C = Hexane/Acetone (1/1)
D = Methyl t-butyl ether
E = Methyl t-butyl ether/Methanol (2/1)

TABLE 2
SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Initial extraction pH	Secondary extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for clean cleanup(mL)	Final extract volume for analysis (mL)
8015DRO	as received	none	none	none		10.0
8081	as received	none	hexane	hexane	10.0	10.0
8100	as received	none	none	cyclohexane	2.0	1.0
8121	as received	none	hexane	hexane	2.0	1.0
8270(c)	as received	none	-	-	-	1.0

(a) To obtain separate acid and base/neutral extracts, Method 3650 should be performed following concentration of the extract to 10.0 mL.

(b) Phenols may be analyzed, by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optical derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.

(c) The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

Table 3.
Sample Preparation Procedures for Soils

Analysis	Aliquots	Surrogates/ Spikes	Surrogate/ Spike Conc.	Sample Weight (grams)	Final Volume (mL)	Exchange Solvent for Analysis	Exchange Solvent for Cleanup	Cleanup Method	Additional Notes
GAM 8015 DRO	3 aliquots MeCl2 100 mLs each	No Surrogates added. 100 ul DRO mix to LCS, MS & MSD	Spike: DRO-2,000 ug/ml	30.0	10.0	none	none	none	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube and concentrate to 10ml.
GAM 8081	3 aliquots hexane 100 mL each. Pest-use 1:1 hexane/ acetone mix	1.0 mL PCB/Pest surrogate 1.0 mL Pest spike to LCS, MS & MSD	Surrogate: PCB-4.0 ug/ml Pest-.40 ug/ml Spike: PCB-100 ug/ml Pest-0.2-0.4 ug/ml	30.0	100 PCB soils 10 pest Soils	hexane for Pest. None for PCB.	none	GCM 3620A, 3665,3660	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube and concentrate to 10ml. Perform hexane exchange for Pest(see hexane exchange in PCB notebook). For PCB, concentrate to 100 ml.
GAM 8100	N/A	0.5 mL PNA surrogate 0.5 mL PNA spike to LCS, MS & MSD	Surrogate: 200 µg/mL Spike: 200 µg/mL	15.0	5	none	Cyclohexane	GCM 3630	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube. Concentrate to 10 mL
GAM 8121	3 aliquots MeCl2 100 mL each	1.0 mL surrogate 1.0 mL spike to LCS, MS & MSD	Surrogate: 10 µg/mL Spike: 0.05/0.50/5.0 µg/mL	30.0	1	hexane	hexane	GCM 3620	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube. Concentrate to 1 mL.

Use detergent washed glassware. Ext

8270		Acid/BN surrogate 1.0 mL Acid/BN spike to LCS, MS & MSD	Acid-75µg/mL BN-50ug/ml Spike+SURR Acid SP 75ug/ml BN SP 50ug/ml Acid surr-37.5 ug/ml BN surr-25 ug/ml						through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube. Concentrate to 10 mL. .
TPH IR 418.1	3 aliquots Freon 30 mL each	No surrogate added . 0.5 mL TPH IR spike to LCS, MS & MSD	Spike: 6,008 ug/ml	30.0	100	none	none	None	Aliquots collected into pre-rinsed funnel with S/P filter paper (grade 413) which funnels into a 100 ml flask containing 3 grams silica gel.

Attachment QAPP-B11

Acid Digestion of Sediments, Sludges & Soils
Method 3050B

GAM 3050B
ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS
Revision 5.0: 2/08/2002

ANALYTES:

		CAS #
Aluminum	(Al)	7429-90-5
Antimony	(Sb)	7440-36-0
Arsenic	(As)	7440-38-2
Barium	(Ba)	7440-39-3
Beryllium	(Be)	7440-41-7
Cadmium	(Cd)	7440-43-9
Calcium	(Ca)	7440-70-2
Chromium	(Cr)	7440-43-9
Cobalt	(Co)	7440-48-4
Copper	(Cu)	7440-50-8
Iron	(Fe)	7439-89-6
Lead	(Pb)	7439-92-1
Magnesium	(Mg)	7439-95-4
Manganese	(Mn)	7439-96-5
Molybdenum	(Mo)	7439-98-7
Nickel	(Ni)	7440-02-0
Potassium	(K)	7440-09-7
Selenium	(Se)	7782-49-2
Silver	(Ag)	7440-22-4
Sodium	(Na)	7440-23-5
Thallium	(Tl)	7440-28-0
Vanadium	(V)	7440-62-2
Zinc	(Zn)	7440-66-6

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by flame or furnace atomic absorption spectroscopy (FLAA and GFAA, respectively) or by inductively coupled argon plasma spectroscopy (ICP-AES) or inductively coupled plasma mass spectroscopy (ICP-MS). Samples prepared by this method may be analyzed by ICP-AES for all the listed elements as long as the detection limits are adequate for the required end-use data. Alternative determinitive techniques may be used if they are scientifically valid and the QC criteria of the method, including those dealing with interferences, can be achieved.

2.0 SUMMARY OF METHOD

2.1 For the digestion of samples for GFAA or ICP-MS analysis, a representative 0.5 gram (wet weight) sample is digested in nitric acid and hydrogen peroxide, then heated for two hours. For FLAA or ICP-AES analysis, 5 ml of conc. HCl is added to the digestate. After refluxing for 15 minutes, the sample is made to volume and analyzed.

3.0 INTERFERENCES

3.1 Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether Method 3050B is applicable to a given waste.

4.0 APPARATUS AND MATERIALS

4.1 Class A volumetric flasks: 5-, 10-, 25-, 50-, 100-, 500- and 2,000mL.

4.2 Varian Vista-MPX ICP-OES, and Ultramass 700 ICP-MS.

4.3 Environmental Express(E.E.) Hot Block digestion unit.

4.4 E.E.ribbed watch glasses.

4.5 Environmental Expess 70 ml digestion vessels w/5mL increment markers.

4.6 Environmental Express push through trace pure teflon filter/plunger units .

4.7 MLA Precision air displacement pipetters: 10-, 25-, 50-, (2)100-, 250-, and 1000-uL. And MLA variable pipetters: 200-1000, and 10-50-uL or equivalent. Repipetter II variable dispensors or equivalent.

4.8 Analytical balance- capable of accurate measurement to 0.01g.
(Mettler-Toledo PB602 (610g max))
Analytical balance- capable of accurate measurement to 0.0001g.
(Sartorious A200S)

4.9 Boekel oven model 107801.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.(Omni*Trace* EM Science cat.no. HXO607-2 from VWR).

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mLs water and dilute to 1 liter in a repipet II liquid dispenser.

5.1.3 Nitric acid (conc), HNO3.(Omni*Trace* EM Science cat.no.NX0407-2 From VWR).

5.1.4 Nitric acid (1:1), HNO3. Add 500 mL concentrated HNO3 to 400 mLs water and dilute to 1 liter in a repipet II liquid dispenser.

5.1.5 Hydrogen peroxide (30%). (Ultrex JT Baker cat.no.JT4155-01 from VWR).

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water must meet ASTM type II standards. Reagent water will be interference free. Reagent water is dispensed from a Barnstead Nano-Pure unit Model #D4741, Serial#

687920145027. Cartridges are changed approximately every 6-8 months, or when 18mΩ or greater cannot be maintained. The Barnstead unit is fed water from a Kinetico commercial reverse osmosis unit (serial #361001). This unit has the capability to produce 75 gallons of water per day and has a 20 gallon bladder tank for storage. This unit also has a two 10", 5 micron prefilter which is changed every other time the cartridges are changed.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous samples shall be preserved to pH <2 with nitric acid and have a holding time of 6 months for analysis of all metals except mercury which has a holding time of 28 days. Soil samples have a holding time of 6 months for all metals except mercury which has a holding time of 28 days.

6.2 Non-Aqueous samples shall be refrigerated at 4°C ± 2° and have a 6 month hold time except for mercury which has a hold time of 28 days.

7.0 PROCEDURE

7.1 Turn on the Environmental Express hot block digestion unit. This takes about 20-30 minutes to reach the desired temperature of 95°C.

7.2 Weigh out a 0.5 gram representative sample in a digestion cup and record the weight in metals preparation log book. All matrix spikes, duplicates, control samples and blanks should be prepared by following the same procedures as the samples.

7.3 Add 5 ml of 1:1 HNO_3 from the individually labeled fixed volume repipet dispensers located in the digestion hood and cover with a watch glass.

7.4 Place the vessel(s) into the hot block unit, at a temperature of 95°C, for a period of 10-15 minutes, allowing the sample to reflux but not allowing it to boil.

7.5 Remove sample from the hot block and allow it to cool in the hood.

7.6 Add 2.5 mLs of concentrated HNO3, replace the watch glass, and reflux for 30 minutes.

7.7 Remove sample from the hot block and allow it to cool in the hood.

7.8 Repeat step 7.6 to ensure complete oxidation. If brown fumes are generated, continue adding conc. HNO_3 in 2.5 mL increments until no brown fumes are given off.

7.9 Heat the sample for two hours at 95°c, ensuring the sample does not boil or go below 5 mLs.

7.10 Remove sample from the hot block and allow it to cool in the hood.

7.11 Add 1 ml of deionized water, from the polyethylene variable volume dispenser, and 1.5 ml of 30% H_2O_2 from the fixed volume repipet dispenser stored in the metals refrigerator, and place the samples into the hot block digestion unit. At this time, the peroxide reaction will take place. You must ensure that excessive reaction does not occur allowing any loss of sample from the vessels. When the reaction subsides, remove the vessels from the hot block and allow them to cool in the hood.

7.12 Add 0.5 ml of H_2O_2 and again place them into the hot block allowing the reaction to go through the cycle again. After each addition of H_2O_2 the beaker must be replaced with its original watch glass, making sure no sample is lost or contaminated.

7.13 Remove sample from the hot block and allow it to cool in the hood.

7.14 Repeat this step until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than 5 ml of H_2O_2 to the sample.

7.15 Continue heating the digestate at 95°c for two hours without boiling, ensuring the volume does not go below 5 mLs.

7.16 For ICP-MS analysis, cool the sample and bring to 50 mL final volume with D.I. water. If necessary, filter the sample using the Env. Express filtermate.

7.17 For the analysis of samples for FLAA or ICP-AES, add 5 ml of concentrated HCl from the fixed volume repipet dispenser, and place on hot block for an additional 15 minutes, again allowing the sample to reflux but not allowing it to boil.

7.18 Remove sample from the hot block and allow it to cool in the hood.

7.19 Rinse watch glass into the sample using deionized water from a squeeze bottle and bring to a final volume of 50 ml.

7.20 Filter with filtermate if necessary.

8.0 QUALITY CONTROL

8.1 Preparation blanks, laboratory control samples (LCS), matrix spikes

(MS)and matrix spike duplicates (MSD) are performed on each analytical batch or 20 samples whichever is more frequent. Associated methods will dictate spiking concentrations.

8.2 CORRECTIVE ACTIONS

8.2.1 Samples associated with control samples and blanks that fall outside of the acceptance criteria of the associated method must be re-digested.

8.2.2 Samples associated with matrix spikes and duplicates that fall outside of the acceptance criteria of the associated method must be flagged as "estimated concentration".

8.2.2 Samples must be re-digested if they boil or are evaporated to dryness.

Attachment QAPP-B12

Separatory Funnel Liquid-Liquid Extraction
Method 3510B

GAM 3510B

Separatory Funnel Liquid-Liquid Extraction
Revision 3.0 2/4/02

1.0 SCOPE AND APPLICATION

1.1 This method describes a procedure for isolating organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for the appropriate determinative methods.

1.2 This method is applicable to the isolation and concentration of water insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures.

2.0 SUMMARY OF METHOD

2.1 A measured volume of sample, usually 1 liter, at a specified pH (see Table 1), is serially extracted with methylene chloride using a separatory funnel. The extract is dried with Na_2SO_4, concentrated(if necessary), and, as necessary, exchanged into a solvent compatible wlth the cleanup or determinative method to be used (see Table 1 for appropriate exchange solvents).

3.0 INTERFERENCES

3.1 Method specific see specific analytical method.

3.2 All glassware must be cleaned according to organic specifications and pre-rinsed with the extraction solvent prior to proceeding with the extraction to minimize organic interferences.

3.3 Phthalate esters are commonly used as plasticizers and present a potential source of contamination. All plastic materials, other than Teflon, should be avoided in the preparation process.

3.4 Some interferences may be present in the samples and may be minimized or removed using the appropriate cleanup procedure.

3.5 Methods 3520/8270, 3510/8270, and 3510/8250, respectively, are preferred over Method 3520/8250 for the analysis of these classes of compounds.

4.0 APPARATUS AND MATERIALS

4.1 Separatory funnel - 2 liter, with Teflon stopcock.

4.2 Drying column - 20 mm ID Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. **Prerinse packed column with 50 mls of solvent used in extraction procedure.**

4.3 Zymark Turbovap, six position with water bath.

4.3.1 200 mL Turbovap tubes w/ 1 mL reservoir tip.

4.3.2 six position tube rack.

4.4 Vials - 2 mL, glass with Teflon lined screw-caps or crimp tops.

4.5 pH indicator paper - pH range including the desired extraction pH.

4.6 Beaker - 250 mL.

4.7 Syringe - 1 mL.

4.8 Pateur glass pipets-1mL, disposabable.

4.9 Glass scintillation vials-20mL, with Teflon or aluminum foil lined screw caps.

4.10 Graduated cylinder - 1 liter.

4.11 Kuderna-Danish (K-D) apparatus.

4.11.1 Concentrator tube - 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.

4.11.2 Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

4.11.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

4.11.4 Snyder column - Two ball micro (Kontes K-569001-0219 or equivalent).

4.11.5 Plastic clips - 1/2 inch (Kontes K-662750 or equivalent).

4.11.6 Boiling chips - Solvent extracted, approximately 10/40 mesh (Teflon or equivalent).

4.11.7 Water bath - Heated, with concentric ring cover, capable of temperature control (+/- 5-C). The bath should be used in a hood.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, can be defined for semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferent is not

observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

5.3 Sodium hydroxide solution (10N), NaOH. Dissolve 40 g NaOH in organic free reagent water and dilute to 100 mL.

5.4 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400-C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

5.5 Sulfuric acid solution (1:1 v/v), H_2SO_4., Slowly add 50 mL of H_2SO_4 to 50 mL of organic-free reagent water.

5.6 Extraction/exchange solvents

5.6.1 Methylene chloride, CH_2Cl_2 - Pesticide quality or equivalent.

5.6.2 Hexane, C_6H_{14} - Pesticide quality or equivalent.

5.6.3 Cyclohexane, C_6H_{12} - Pesticide quality or equivalent.

5.6.4 Freon 113, fluorocarbon-113 (1,1,2-trichloro-1,2,2-trifluoroethane): Mid-Atlantic Chemical.

5.7 Surrogate and Matrix Spiking Solutions (see Table 2 for appropriate volumes added for each method.)

5.7.1 8015 DRO Spiking Mix - DRO mix 2,000µg/mL Restek cat. no. 31064.

5.7.2 418.1 - Spike solution (Accustandard cat. no. M-418) neat components in the following parts by volume: Chlorobenzene 10.0, n-Hexadecane 15.0, Isooctane 15.0.

5.7.3 8081 Pesticide Matrix Spike Mix, Restek cat. no. 32018. 25-50 ug/ml.

5.7.4 8081 Pesticide Surrogate Mix, Restek cat. no. 32000. 200 ug/ml.

5.7.5 8100 Spike Mix, Restek cat. no. 31011 diluted to 200 ug/mL in acetone.

5.7.6 8100 Surrogate Mix - 2-Fluorobiphenyl 99%(Aldrich catalog #10,274-1)and ortho-Terphenyl 99% (cat# T280-0)to a concentration of 200 ug/mL.

5.7.7 8121 Spike Mix: AccuStandard cat# 8121M Diluted to 0.05/0.50/5.0 (same concentration as initial calibration point #3 from table 4, GAM 8121.

5.7.8 1000 mg/L of 1,4-dichloronaphthalene and dilute it to 10 µg/uL.

5.7.9 8270 Matrix Spike Mix - Acid Compounds at 15 µg/mL and Base/Neutral Compounds at 10 µg/mL.

5.7.10 8270 Surrogate standards - Acid surrogates (RESTEK #31087) phenol-d(6), 2-fluorophenol, 2,4,6-tribromophenol at 15 µg/mL. Base Neutral surrogates (RESTEK #31086) nitrobenzene-d(5), 2-fluorobiphenyl, and p-terphenyl-d(14) at 10 µg/mL.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Water samples are collected in one liter or larger glass containers with Teflon lined lids.

6.2 Samples that are suspected of containing residual chlorine should be preserved with 10% $Na_2S_2O_3$/gallon at the time of collection.

7.0 PROCEDURE

7.1 Using a 1 liter graduated cylinder, measure 1 liter (nominal) of sample and transfer it quantitatively to the separatory funnel. If high concentrations are anticipated, a smaller volume may be used and then diluted with organic-free reagent water to 1 liter. **Add 1.0 mL of the surrogate standards to all samples, spikes, and blanks**. Refer to Table 2 for each determinative method to be used, for details on the surrogate standard solution and the matrix spike solution). For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 10 ug/mL of each base/neutral analyte and 15 ug/mL of each acid analyte in the extract to be analyzed.

7.1.1 For each analytical batch or 20 samples, a preparation blank, laboratory control sample (LCS), matrix spike (MS) and matrix spike duplicate must be processed.

7.1.2 Refer to table 2 for the appropriate volume of Spiking solution for each specific semivolatiles method being used.

7.2 Check the pH of the sample with wide-range pH paper and, if necessary, adjust the pH to that indicated in Table 1 for the specific determinative method that will be used to analyze the extract.

7.3 Add 60 mL of methylene chloride to the separatory funnel.

7.4 Seal and shake the separatory funnel vigorously for 1-2 minutes with periodic venting to release excess pressure.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. Venting of the separatory funnel should be into a hood to avoid needless exposure of the analyst to solvent vapors.

7.5 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken (recovery of < 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in GAM 3520, Continuous Liquid/Liquid Extraction.

7.6 Repeat the extraction two more times using fresh portions of solvent(Sections 7.3 through 7.5). Combine the three solvent extracts.

7.7 If further pH adjustment and extraction is required, adjust the pH of the aqueous phase to the desired pH indicated in Table 1. Serially extract three times with 60 mL of methylene chloride, as outlined in Sections 7.3 through 7.5. Collect and combine the extracts and label the combined extract appropriately.

7.8 If performing GC/MS analysis (GAM 8270), the acid/neutral and base extracts may be combined prior to concentration. However, in some situations, separate concentration and analysis of the acid/neutral and base extracts may be preferable (e.g., if for regulatory purposes the presence or absence of specific acid/neutral are base compounds at low concentrations must be determined, separate extract analyses may be warranted).

7.9 Perform the concentration (if necessary) using the Nitrogen Blowdown Technique (7.10)using the Turbovap.

7.10 Nitrogen Blowdown Technique

7.10.1 Place the concentrator tube in a warm bath (35-C) and evaporate the solvent volume to 0.5 mL using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: New plastic tubing must not be used between the carbon trap and the sample, since it may introduce interferences.

7.10.2 The internal wall of the tube must be rinsed down several times with methylene chloride or appropriate solvent during the operation. During evaporation, the tube solvent level must be positioned to avoid water condensation. Under normal procedures, the extract must not be allowed to become dry. CAUTION: When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost.

7.11 The extract may now be analyzed for the target analytes using the appropriate determinative technique(s). If analysis of the extract will not be performed immediately, stopper the concentrator tube and store refrigerated. If the extract will be stored longer than 2 days it should be transferred to a vial with a Teflon lined screw-cap or crimp top, and labeled appropriately. Extracts must be stored at 4° C until analysis.

7.12 K-D Technique

7.12.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporation flask. Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20-30 mL of methylene chloride and add it to the column to complete the quantitative transfer.

7.12.2 Add one or two clean boiling chips to the flask and attach a three ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D

apparatus on a hot water bath (15-20-C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10-20 minutes. At the proper rate of distillation

the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

7.12.3 If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add 50 mL of the exchange solvent, a new boiling chip, and reattach the Snyder column. Concentrate the extract, as described in Section 7.11, raising the temperature of the water bath, if necessary, to maintain proper distillation.

7.12.4 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Section 7.11 or adjusted to 10.0 mL with the solvent last used. Extracts must be stored at 4° C until analysis.

7.13 If further concentration is indicated in Table 1, either the microSnyder column technique (7.13.1) or nitrogen blowdown technique (7.10) is used to adjust the extract to the final volume required.

7.13.1 Micro-Snyder Column Technique

7.13.1.1 If further concentration is indicated in Table 1, add another clean boiling chip to the concentrator tube and attach a two ball micro-Snyder column. Prewet the column by adding 0.5 mL of methylene chloride or exchange solvent to the top of the column. Place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 5-10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 0.2 mL of extraction solvent. Adjust the final volume to 1.0-2.0 mL, as indicated in Table 1, with solvent. Extracts must be stored at 4° C until analysis.

8.0 QUALITY CONTROL

8.1 Any reagent blanks or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to Chapter One for specific quality control procedures and Method for extraction and sample preparation procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

10.0 REFERENCES

10.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

TABLE 1. SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Initial extraction pH	Secondary extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for clean cleanup(mL)	Final extract volume for analysis(mL)
8015 DRO	as received	none	none	-	-	10.0
8081	5-9	none	hexane	hexane	10.0	10.0
8100	as received	none	none	cyclohexane	2.0	1.0
8121	as received	none	hexane	hexane	2.0	1.0
8270(bd)	<2	>11	none	-	-	1.0

(a) Phenols may be analyzed, by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optional derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.

(b) The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

(c) Loss of phthalate esters, organochlorine pesticides and phenols can occur under these extraction conditions (see Section 3.2).

(d) Extraction pH sequence may be reversed to better separate acid and neutral waste components. Excessive pH adjustments may result in the loss of some analytes (see Section 3.2).

Table 2.
Sample Preparation Procedures for Water

Analysis	Aliquots	Surrogates/ Spikes	Surrogate/ Spike Conc.	Initial Volume (L)	Initial extraction pH	Secondary extraction pH	Final Volume (ml)	Exchange Solvent for Analysis	Exchange Solvent for Cleanup	Cleanup Method	Additional Notes
GAM 8015 *(DRO)*	3 aliquots *MeCl2 60ml. ea.*	No surrogate *added. 200 ul* DRO Spike to LCS & LCSD	Spike: DRO- 2,000 ug/ml	1	as *received*	none	10	none	none	none	Aliquots collected into beaker. Pour through pre-*rinsed glass column with bed of* Na2SO4 and glass wool. Collect into Zymark.
GAM 418.1	3 aliquots Freon 30ml. ea.	No surrogate added . 1.0 mL TPH IR spike to LCS & LCSD	Spike: 6,008ug/ml	1	<2 1:1 HCl	none	100	none	none	none	Aliquots collected into pre-rinsed funnel with S/P filter paper (grade 413). Pour into a 100 ml volumetric flask containing 3g. silica gel.
GAM 8100	3 aliquots MeCl2 60ml. ea.	1.0 mL PNA surrogate 1.0 mL PNA spike to LCS & LCSD	Surrogates: 200 ug/mL Spike: 200 ug/mL	1	as received	none	10	none	cyclohexane	3630	Use detergent washed glassware. Aliquots collected into beaker. Pour through prerinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube.
GAM 8121	3 aliquots MeCl2 60ml. ea.	1.0 mL of 1,4-Dichloro-naphthalene 1.0 mL of spike mix to LCS & LCSD	Surrogates: 10.0 ug/mL Spike: 0.05/0.50/5.0 ug/mL	1	as received	none	1	hexane	hexane	3620	
GAM 8270	3 aliquots MeCl2 60ml. ea.	1.0 mL Acid/BN surrogate 1.0 mL Acid/BN spike to LCS & LCSD	Surrogates: Acid-15 ug/mL BN-10 ug/mL Spike: Acid-15 ug/mL BN- 10 ug/mL	1	<2 1:1 H2SO4	>11 10N NaOH	1	none	-	-	Use detergent washed glassware. Acid fraction first: Collect into beaker. Pour through prerinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube. Blow down to 10mls. B/N fraction: Adjust pH to 14. Follow same procedure as acid fraction. Collect into Zymark tube containing 10mls. from acid fraction.
GAM 8081	3 aliquots MeCl2 60ml. ea.	1.0 mL PCB/Pest surrogate 1.0 ml PCB spike, 1.0 mL Pest spike to LCS & LCSD	Surrogates: 0.4 ug/ml Spike: 0.2-0.4 ug/ml-PEST 10.0 ug/ml - PCB	1	<2 1:1 H2SO4	none	10	hexane	hexane	3620 3665 3660	Use detergent washed glassware. Collect into beaker. Pour through prerinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube and blow down to 1ml. Perform hexane exchange (see hexane exchange in PCB notebook).

GAM 8270 TCLP as received	3 aliquots MeCl2 60ml. ea.	1.0 mL Acid/BN surrogate 1.0 mL TCLP Acid/BN spike to LCS, MS & MSD	Surrogates: Acid-15 ug/ml BN-10 ug/ml Spike: 10 ug/ml	100ml sample 900ml Deionized H2O	<2 1:1 H2SO4	>11 10N NaOH	1	none	-		Use detergent washed glassware. Collect into beaker. Pour through preinsed glass column with bed of Na2SO4 and glass wool. Collect into Zymark tube and blow down to 10mls. B/N fraction: Follow same procedure as acid fraction. Collect into Zymark tube containing 10mls. from acid fraction.

Attachment QAPP-B13

Pressurized Fluid Extraction (PFE)
Method 3545A

GAM 3545A
PRESSURIZED FLUID EXTRACTION (PFE)
Revision 1.0 02/05/02

1.0 SCOPE AND APPLICATION

1.1 Method 3545 is a procedure for extracting water insoluble or slightly water soluble semivolatile organic compounds from soils, clays, sediments, sludges, and waste solids. The method uses e levated temperature(100EC) and pressure (1500 - 2000 psi)to achieve analyte recoveries equivalent to those from Soxhlet extraction, using less solvent and taking significantly less time than the Soxhlet procedure. This procedure was developed and validated on a commercially-available, automated extraction system.

1.2 This method is applicable to the extraction of semivolatile organic compounds,organophosphorus pesticides, organochlorine pesticides, chlorinated herbicides, and PCBs, which may then be analyzed by a variety of chromatographic procedures.

1.3 This method has been validated for solid matrices containing 250 to 12,500 µg/kg of semivolatile organic compounds, 250 to 2500 µg/kg of organophosphorus pesticides, 5 to 250 µg/kg of organochlorine pesticides, 50 to 5000 µg/kg of chlorinated herbicides, and 1 to 1400 µg/kg of PCBs. The method may be applicable to samples containing these analytes at higher concentrations and may be employed after adequate performance has been demonstrated for the concentrations of interest (see Method 3500, Sec. 8.0).

1.4 This method is applicable to solid samples only, and is most effective on dry materials with small particle sizes. Therefore, waste samples must undergo phase separation, as described in Chapter Two, and only the solid phase material is to be extracted by this procedure. If possible, soil/sediment samples may be air-dried and ground to a fine powder prior to extraction. Alternatively, if the loss of analytes during drying is a concern, soil/sediment samples may be mixed with anhydrous sodium sulfate or pelletized diatomaceous earth. The total mass of material to be prepared depends on the specifications of the determinative method and the sensitivity required for the analysis, but 10 - 30 g of material are usually necessary and can be accommodated by this extraction procedure.

1.5 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 A 15g sample is prepared for extraction either by air drying the sample, or by mixing the sample with 15 g of anhydrous sodium sulfate or pelletized diatomaceous earth. The sample is then ground to a 100 - 200 mesh powder (150 µm to 75 µm) and loaded into the extraction cell.

2.2 The extraction cell containing the sample is heated to the extraction temperature (see Sec. 7.8), pressurized with the appropriate solvent system, and extracted for 5 minutes (or as recommended by the instrument manufacturer). The solvent systems used for this procedure vary with the analytes of interest and are described in Sec. 5.5.

2.3 The solvent is collected from the heated extraction vessel and allowed to cool. Since the extraction cells contain frits, no filtration of the extracts is needed. However, the extraction of very wet samples (e.g., 30% moisture) may require that the extract be dried with additional pelletized diatomaceous earth.

2.4 The extract may be concentrated, if necessary, and, as needed, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

3.2 If necessary, Florisil and/or sulfur cleanup procedures may be employed. In such cases, proceed with Method 3620 and/or Method 3660.

3.3 All glassware must be cleaned according to organic specifications and pre-rinsed with the extraction solvent prior to proceeding with the extraction to minimize organic interferences.

3.4 Phthalate esters are commonly used as plasticizers and present a potential source of contamination. All plastic materials, other than Teflon, should be avoided in the preparation process.

4.0 APPARATUS AND MATERIALS

4.1 Pressurized fluid extraction device

4.1.1 Dionex Accelerated Solvent Extractor or Supelco SFE-400 with appropriately-sized extraction cells. Currently, cells are available that will accommodate 10-g, 20-g and 30g samples. Cells should be made of stainless steel or other material capable of withstanding the pressure requirements (2000+ psi) necessary for this procedure.

4.1.2 Other system designs may be employed, provided that adequate performance can be demonstrated for the analytes and matrices of interest.

4.2 Apparatus for determining percent dry weight

4.2.1 Oven - drying

4.2.2 Desiccator

4.2.3 Crucibles -porcelain or disposable aluminum

4.3 Apparatus for grinding - capable of reducing particle size to < 1mm.

4.4 Analytical balance - capable to weighing to 0.01 g.

4.5 Vials for collection of extracts - 40-mL or 60-mL, pre-cleaned, open top screw-cap with PTFE-lined silicone septum (Dionex 049459, 049460, 049461, 049462 or equivalent).

4.6 Filter disk - 1.91 cm, Type D28 (Whatman 10289356, or equivalent).

4.7 Cell cap sealing disk (Dionex 49454, 49455, or equivalent).

4.8 Pasteur glass pipettes-1mL, disposable.

4.9 Beakers-250mL.

4.10 Turbovap tube racks 6 position.

4.11 Vials-2mL, for GC autosampler, with Teflon lined screw caps or crimp tops.

4.12 Glass scintillation vials-20mL, with Teflon or aluminum lined screw caps.

4.13 Spatula-stainless steel or Teflon.

4.14 Drying column-300mm by 20mm ID Pyrex Chromatographic column with Pyrex glass wool at bottom and Teflon stopcock. Use a small pad of Pyrex glass wool to retain the absorbent. **Pre-rinse packed column with 50mL of solvent used in extraction.**

4.15 Syringe-1mL

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Drying agents

5.3.1 Sodium sulfate (granular anhydrous), Na_2SO_4.

5.3.2 Pelletized diatomaceous earth.

5.3.3 The drying agents should be purified by heating at 400 C for 4 hours in a shallow tray, or by extraction with methylene chloride. If extraction with methylene chloride is employed, then a reagent blank should be prepared to demonstrate that the drying agent is free of interferences.

5.4 Phosphoric acid solution (see Sec. 5.5.5). Prepare a 1:1 (v/v) solution of 85% phosphoric acid (H_3PO_4) in organic-free reagent water.

5.5 Extraction solvents. The extraction solvent to be employed depends on the analytes to be extracted, as described below. All solvents should be pesticide quality or equivalent. Solvents may be degassed prior to use.

5.5.1 Organochlorine pesticides may be extracted with acetone/hexane (1:1, v/v), CH_3COCH_3 /C_6H_{14} or acetone/methylene chloride (1:1,v/v), CH_3COCH_3 /CH_2Cl_2.

5.5.2 Semivolatile organics may be extracted with acetone/methylene chloride (1:1,v/v), CH_3COCH_3 /CH_2Cl_2 or acetone/hexane (1:1, v/v), CH_3COCH_3 /C_6H_{14}

5.5.3 PCBs may be extracted with acetone/hexane (1:1, v/v), CH_3COCH_3 /C_6H_{14} acetone/methylene chloride (1:1, v/v), CH_3COCH_3 /CH_2Cl_2 or hexane, C_6H_{14}

5.5.4 Organophosphorus pesticides may be extracted with methylene chloride, CH_2Cl_2 or acetone/methylene chloride (1:1, v/v), CH_3COCH_3 /CH_2Cl_2.

5.5.5 Chlorinated herbicides may be extracted with an acetone/methylene chloride/phosphoric acid solution (250:125:15, v/v/v), CH_3COCH_3/ CH_2Cl_2/H_3PO_4, or an acetone/methylene chloride/trifluoroacetic acid solution (250:125:1, v/v/v), CH_3COCH_3/ CH_2Cl_2/CF_3COOH. (If the second option is used, the trifluoroacetic acid solution should be prepared by mixing 1% trifluoroacetic acid in acetonitrile.) Make fresh solutions before each batch of extractions.

5.5.6 Other solvent systems may be employed, provided that the analyst can demonstrate adequate performance for the analytes of interest in the sample matrix (see Method 3500, Sec. 8.0). CAUTION: For best results with very wet samples (e.g., 30% moisture), reduce or eliminate the quantity of hydrophilic solvent used.

5.6 High-purity gases such as nitrogen, carbon dioxide, or helium are used to purge and/or pressurize the extraction cell. Follow the instrument manufacturer's recommendation for the choice of gases.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analysis, Sec. 4.1.

6.2 Soil samples are collected in 125mL or 250mL wide mouth precleaned glass containers with Teflon lined lids. Refrigerated to 4°C from time of collection to sample analysis.

7.0 PROCEDURE

7.1 Sample preparation

7.1.1 Sediment/soil samples - Decant and discard any water layer on sediment samples. Mix the sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks. If sample is still wet, additional drying agent may need to be added. Alternatively, mix the sample with an equal volume of anhydrous sodium sulfate or pelletized diatomaceous earth until a free-flowing powder is obtained. NOTE: Dry, finely ground soil/sediment allows the best extraction efficiency for nonvolatile, non-polar organics, e.g., 4,4'-DDT, PCBs, etc. Air-drying may not be appropriate for the analysis of the more volatile organochlorine pesticides (e.g., the BHCs) or the more volatile of the semi-volatile organics because of losses during the drying process. The use of sodium sulfate as a drying agent can lead to clogging of the frits in the cell with re-crystallized sodium sulfate. (See "Caution" following Sec. 5.5.6.)

7.1.2 Waste samples - Multiphase waste samples must be prepared by the phase separation method in Chapter Two before extraction. This extraction procedure is for solids only.

7.1.3 Dry sediment/soil and dry waste samples amenable to grinding. Grind or otherwise reduce the particle size of the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Disassemble grinder between samples, according to manufacturer's instructions, and decontaminate with soap and water, followed by acetone and hexane rinses. NOTE: The note in Sec. 7.1.1 also applies to the grinding process.

7.1.4 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The analyst may add anhydrous sodium sulfate, pelletized diatomaceous earth, sand, or other clean, dry reagents to the ample to make it more amenable to grinding.

7.2 Determination of percent dry weight - When sample results are to be calculated on a dry weight basis, a second portion of sample should be weighed at the same time as the portion used for analytical determination. WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated sample.% dry weight ' g of dry sample g of sample x 100.

7.2.1 Immediately after weighing the sample for extraction, weigh 5-10g of the sample into a tared crucible or beaker. Dry this aliquot overnight at 105 C. Allow to cool in a desiccator before weighing. Calculate the % dry weight as follows:

7.3 Grind a sufficient weight of the dried sample from Sec. 7.1 to yield the sample weight needed for the determinative method (usually 10 - 30 g). Grind the sample until it passes through a 10 mesh sieve.

7.4 Transfer the ground sample to an extraction cell of the appropriate size for the aliquot. Generally, an 11-mL cell will hold about 10 g of material, a 22-mL cell will hold about 20 g of material, and a 33-mL cell will hold about 30 g of material. The weight of a specific sample that a cell will contain depends on the bulk density of the sample and the amount of drying agent that must be added to the sample in order to make it suitable for extraction. Analysts should ensure that the sample aliquot extracted is large enough to provide the necessary sensitivity and choose the extraction cell size accordingly. Use disposable cellulose or glass fiber filters in the cell outlets. Clean sand may be used to fill any void volume in the extraction cells.

7.5 Add the surrogates listed in the determinative method to each sample. Add the matrix spike/matrix spike duplicate compounds listed in the determinative method to the two additional aliquots of the sample selected for spiking.

7.5.1 For each analytical batch or 20 samples, a preparation blank, laboratory control sample (LCS), matrix spike (MS) and matrix spike duplicate (MSD) must be processed.

7.5.2 Refer to Table 1 for the appropriate volume of spiking solution for each specific semi-volatiles method being used.

7.6 Place the extraction cell into the instrument or autosampler tray, as described by the instrument manufacturer.

7.7 Place a pre-cleaned collection vessel in the instrument for each sample, as described by the instrument manufacturer. The total volume of the collected extract will depend on the specific instrumentation and the extraction procedure recommended by the manufacturer and may range from 0.5 to 1.4 times the volume of the extraction cell. Ensure that the collection vessel is sufficiently large to hold the extract.

7.8 Recommended extraction conditions
Oven temperature: 100°C
Pressure: 1500 - 2000 psi
Static time: 5 min (after 5min pre-heat equilibration)
Flush volume: 60% of the cell volume
Nitrogen purge: 60 sec at 150 psi (purge time may be extended for larger cells)
Static Cycles: 1

7.8.1 Optimize the conditions, as needed, according to the manufacturer's instructions. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample. Any pressure in the range of 1500 - 2000 psi should suffice.

7.8.2 Once established, the same pressure should be used for all samples extracted for the same analysis type.

7.9 Begin the extraction according to the manufacturer's instructions.

7.10 Collect each extract in a clean vial (see Sec. 7.7). Allow the extracts to cool after the extractions are complete.

7.11 Pour each sample through a pre-rinsed 300mm x 20mm drying column. Rinse vial twice with extraction solvent and pour through the column. Sample is collected in a Zymark Turbovap concentrator tube. Extract must be stored at 4°C until analysis.

7.12 The extract is now ready for concentration, cleanup, or analysis, depending on the extent of interferents and the determinative method to be employed. Refer to Method 3600 for guidance on selecting appropriate cleanup methods. Excess water present in extracts may be removed by filtering the extract through a bed of anhydrous sodium sulfate. Certain cleanup and/or determinative methods may require a solvent exchange prior to cleanup and/or sample analysis.

7.13 Nitrogen Blow Down Technique

7.13.1 Place the concentrator tube in a warm water bath (Approx. 43°C). Evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

7.13.2 The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During the evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath.) Under normal operating conditions, the extract should not be allowed to become dry. Extract must be stored at 4°C.

CAUTION: When the volume of solvent reduced below 0.5mL, semi-volatile analytes may be lost.

7.14 If the phosphoric acid solution in Sec. 5.5.5 is used for the extraction of chlorinated herbicides, then the extractor should be rinsed by pumping acetone through all the lines of the system. The use of other solvents for these analytes may not require this rinse step.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One and Method 8000 for guidance on quality control procedures. Refer to Method 3500 for specific guidance on extraction and sample preparation procedures.

8.2 Before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a solid matrix method blank (e.g., clean sand). Each time samples are extracted, and when there is a change in reagents, a method blank needs to be extracted and analyzed for the compounds of interest. The method blank should be carried through all stages of the sample preparation and measurement.

8.3 Standard quality assurance practices should be used with this method. Field duplicates should be collected to validate the precision of the sampling procedures. A matrix spike/matrix spike duplicate, or matrix spike and duplicate sample analysis, and a laboratory control sample should be prepared and analyzed with each batch of samples prepared by this procedure, unless the determinative method provides other guidance.

8.4 Surrogate standards should be added to all samples when listed in the appropriate determinative method.

8.5 Any reagent blanks, matrix spike and laboratory control samples should be subjected to exactly the same analytical procedures as those used on actual samples.

8.6 Refer to the specific method for specific quality control procedures.

9.0 METHOD PERFORMANCE

9.1 Chlorinated pesticides and semi-volatile organics.

Single-laboratory accuracy data were obtained for chlorinated pesticides and semi-volatile organics at three different spiking concentrations in three different soil types. Spiking concentrations ranged from 5 to 250 µg/kg for the chlorinated pesticides and from 250 to 12500 µg/kg for the semi-volatiles. Spiked samples were extracted both by the Dionex Accelerated Solvent Extraction system and by a Perstorp Environmental Soxtec™ (automated Soxhlet). Extracts were analyzed either by Method 8270 or Method 8081. Method blanks, spikes and spike duplicates were included for the low concentration spikes; matrix spikes were included for all other concentrations. The data are reported in detail in Reference 1, and represent seven replicate extractions and analyses for each sample. Data summary tables are included in Methods 8270 and 8081.

9.2 Organophosphorus pesticides and chlorinated herbicides.

Single-laboratory accuracy data were obtained for organophosphorus pesticides (OPPs) and chlorinated herbicides at two different spiking concentrations in three different soil types. Spiking concentrations ranged from 250 to 2500 µg/kg for the OPPs and from 50 to 5000 µg/kg for the chlorinated herbicides. Chlorinated herbicides were spiked with a mixture of the free acid and the ester (1:1). Spiked samples were extracted both by the Dionex Accelerated Solvent Extractor and by Soxhlet for the OPPs. Extracts were analyzed by Method 8141. Spiked chlorinated herbicides were extracted by the Dionex Accelerated Solvent Extractor and by the shaking method described in Method

8151. Extracts were analyzed by Method 8151. Method blanks, spikes and spike duplicates were included for the low concentration spikes; matrix spikes were included for all other concentrations. The data are reported in detail in Reference 2, and represent seven replicate extractions and analyses for each sample. Data summary tables are included in Methods 8141 and 8151.

9.3 PCBs

Single-laboratory accuracy data were obtained for PCBs from a soil sample with PCB content certified by NIST (Standard Reference Material, SRM 1939, River Sediment). A PCB-contaminated soil was purchased from a commercial source. Spiking or certified concentrations ranged from 1 to 1400 µg/kg. Samples were extracted by the Dionex Accelerated Solvent Extractor and by Soxtec™ (Perstorp Environmental). Extracts were analyzed using Method 8082. Method blanks, spikes and spike duplicates were included. The data are reported in Reference 2, and represent seven replicate extractions and analyses for each sample. Data summary tables are included in Method 8082.

10.0 REFERENCES

1. B. Richter, Ezzell, J., and Felix, D., "Single Laboratory Method Validation Report. Extraction of TCL/PPL (Target Compound List/Priority Pollutant List) BNAs and Pesticides using Accelerated Solvent Extraction (ASE) with Analytical Validation by GC/MS and GC/ECD"; Document 116064.A, Dionex Corporation, June 16, 1994.

2. B. Richter, Ezzell, J., and Felix, D., "Single Laboratory Method Validation Report. Extraction of TCL/PPL (Target Compound List/Priority Pollutant List) OPPs, Chlorinated Herbicides and PCBs using Accelerated Solvent Extraction (ASE)". Document 101124, Dionex Corporation, December 2, 1994).

11.0 SAFETY

The use of organic solvents, elevated temperatures, and high pressures in Method 3545 present potential safety concerns in the laboratory. Common sense laboratory practices can be employed to minimize these concerns. However, the following sections describe additional steps that should be taken.

11.1 Extraction cells in the oven are hot enough to burn unprotected skin. Allow the cells to cool before removing them from the oven or use appropriate protective equipment (e.g., insulated gloves or tongs), as recommended by the manufacturer.

11.2 During the gas purge step, some solvent vapors may exit through a vent port in the instrument. Follow the manufacturer's directions regarding connecting this port to a fume hood or other means to prevent release of solvent vapors to the laboratory atmosphere.

11.3 The instrument may contain flammable vapor sensors and should be operated with all covers in place and doors closed to ensure proper operation of the sensors. If so equipped, follow the manufacturer's directions regarding replacement of extraction cell seals when frequent vapor leaks are detected.

Table 1.
Sample Preparation Procedures for Soils

Analysis	Aliquots	Surrogates/ Spikes	Surrogate/ Spike Conc.	Sample Weight (grams)	Final Volume (mL)	Exchange Solvent	Exchange Solvent for Cleanup	Cleanup Method	Additional Notes
GAM 8015 DRO	3 aliquots MeCl2 100 mLs each	No Surrogates added. 100 ul DRO mix to LCS, MS & MSD	Spike: DRO-2,000 ug/ml	30.0	10.0	none	none	none	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube and concentrate to 10ml.
GAM 8081	3 aliquots hexane 100 mL each. Pest-use 1:1 hexane/ acetone mix	1.0 mL PCB/Pest surrogate 1.0 mL Pest spike to LCS, MS & MSD	Surrogate: PCB-4.0 ug/ml Pest-.40 ug/ml Spike: PCB-100 ug/ml Pest-0.2-0.4 ug/ml	30.0	100 PCB soils 10 pest Soils	hexane for Pest. None for PCB.	none	GCM 3620A, 3665,3660	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube and concentrate to 10ml. Perform hexane exchange for Pest(see hexane exchange in PCB notebook). For PCB, concentrate to 100 ml.
GAM 8100	N/A	0.5 mL PNA surrogate 0.5 mL PNA spike to LCS, MS & MSD	Surrogate: 200 µg/mL Spike: 200 µg/mL	15.0	5	none	Cyclohexane	GCM 3630	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube. Concentrate to 10 mL
GAM 8121	3 aliquots MeCl2 100 mL each	1.0 mL surrogate 1.0 mL spike to LCS, MS & MSD	Surrogate: 10 µg/mL Spike: 0.05/0.50/5.0 µg/mL	30.0	1	hexane	hexane	GCM 3620	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube. Concentrate to 1 mL.
GAM 8270	N/A	1.0 mL Acid/BN surrogate 1.0 mL Acid/BN spike to LCS, MS & MSD	Surrogate: Acid-75µg/mL BN-50ug/ml Spike+SURR Acid SP 75ug/ml BN SP 50ug/ml Acid surr-37.5 ug/ml BN surr-25 ug/ml	15.0	5	none	none	-	Use detergent washed glassware. Pour through pre-rinsed glass column with bed of Na2SO4 and glass wool. Collect in Zymark tube. Concentrate to 10 mL.
TPH IR 418.1	3 aliquots Freon 30 mL each	No surrogate added . 0.5 mL TPH IR spike to LCS, MS & MSD	Spike: 6,008 ug/ml	30.0	100	none	none	None	Aliquots collected into pre-rinsed funnel with S/P filter paper (grade 413) which funnels into a 100 ml flask containing 3 grams silica gel.

Attachment QAPP-B14

Florisil Column Cleanup
Method 3620A

GCM 3620A
Florisil Column Cleanup
Revision 2.1: 01/10/97

1.0 SCOPE AND APPLICATION

1.1 Florisil, a registered trade name of the Floridin Co., is a magnesium silicate with acidic properties. It is used for general column chromatography as a cleanup procedure prior to sample analysis by gas chromatography.

1.2 General applications: Cleanup of pesticide residues and other chlorinated hydrocarbons; the separation of nitrogen compounds from hydrocarbons, the separation of aromatic compounds from aliphatic-aromatic mixtures; and similar applications for use with fats, oils, and waxes (Floridin).

1.3 Specific applications: This method includes guidance for cleanup of sample extracts containing the following analyte groups:; organochlorine pesticides (GAM 8081); chlorinated hydrocarbons (GAM 8121); and diphenylamine (GAM 8270B).

2.0 SUMMARY OF METHOD

2.1 The column is packed with the required adsorbent, topped with a water adsorbent, and then loaded with the sample to be analyzed. Elution is effected with a suitable solvent(s) leaving the interfering compounds on the column. The eluate is then concentrated. Pre-packed cartridges are also used because of the ease and consistency of the media.

3.0 INTERFERENCES

3.1 A reagent blank should be performed for the compounds of interest prior to the use of this method. The level of interferences must be below the method detection limit before this method is performed on actual samples.

3.2 More extensive procedures than those outlined in this method may be necessary for reagent purification.

4.0 APPARATUS AND MATERIALS

4.1 Beaker: 250 and 500 mL.

4.2 Clean-up column: Alltech part # 307950, Extract-Clean RC column 1000mg.

4.3 Concentrator apparatus: Zymark Turbovap with 200 mL tubes.

4.4 Muffle Furnace capable of 350 degrees centigrade. Thermolyne 1300.

4.5 Reagent bottle: 500-mL.

4.6 Boiling chips: Solvent extracted, approximately 10/40 mesh (Teflon). Chemware # A1069103 or equivalent.

4.7 Vacuum manifold: 12 port vacuum manifold (Alltech #210351 or equivalent),consisting of glass vacuum basin, collection rack and

funnel, collection vials, replaceable stainless steel delivery tips, built-in vacuum bleed valve and gauge.

5.0 REAGENTS

5.1 Florisil: Pesticide residue (PR) grade (60/100 mesh); purchase-activated are Florisil cartridges such as Alltech # 207930 or 307950 preloaded
5.2 Sodium sulfate (ACS): Granular, anhydrous. Mallinckrodt # 8024 or equivalent.

5.3 Eluting solvents:

5.3.1 Diethyl ether: Pesticide quality, preserved with 2% ethanol to prevent peroxide formation. Burdick & Jackson # 106-1 or equivalent.

5.3.2 Hexane: Pesticide quality, Burdick & Jackson #217-4 or equivalent.

5.3.3 Petroleum ether (boiling range 30 - 60-C) - Pesticide quality or equivalent.

5.3.4 Pentane, CH3(CH2)3CH3 - Pesticide quality or equivalent.

5.3.5 Acetone, CH3COCH3 - Pesticide quality or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Water and soil samples are stored refrigerated at 4°C. Water samples must be extracted within seven (7) days and soil samples extracted within 14 days of sample collection.

6.2 Extracts must be stored under refrigeration and must be analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Reduce the sample extract volume to 1 mL prior to cleanup. The extract solvent must be hexane. Solvent exchange is performed as follows: Concentrate extract to 1 mL. Add a Teflon boiling chip to the Zymark tube and raise the temperature to the highest setting (about 45 C). Add 10 mLs of hexane to rinse the walls of the tube and blow down to 1 mL. Repeat once more with hexane and take to a final volume of 1 mL (approximate).

7.2 Cartridge Cleanup Techniques

7.2.1 Arrange the 1 g Florisil cartridges on the manifold in the closed-valve position. Other size cartridges may be used, however the data presented in the Tables is all based on 1 g cartridges for pesticides/Aroclors. Therefore, supporting recovery data must be developed for other sizes. Larger cartridges will probably require larger volumes of elution solvents.

7.2.2 Turn vacuum pump on and set pump vacuum to 10 inches or 254 mm of Hg. Do not exceed the manufacturer's recommendation for manifold vacuum. Flow rates can be controlled by opening and closing cartridge valves.

7.2.3 Condition the cartridges by adding 10 mL of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off the vacuum.

7.2.4 Slowly open cartridge valves to allow the hexane to pass through the cartridges. Close the cartridge valves when there is still at least 1 mm of solvent above the sorbent bed. Do not allow cartridges to become dry. If cartridges go dry, repeat the conditioning step.

7.2.5 Transfer the extract to the cartridge. Open the cartridge valve to allow the extract to pass through the cartridge bed at approximately 2 mL/minute.

7.2.6 When the entire extract has passed through the cartridges, but before the cartridge becomes dry, rinse the sample vials with an additional 1 mL of hexane, and add the rinse to the cartridges to complete the quantitative transfer.

7.2.7 Close the cartridge valve and turn off the vacuum after the solvent has passed through, ensuring that the cartridge never goes dry.

7.2.8 Place a 20 mL scintillation vial or test tube into the sample rack corresponding to the cartridge position. Attach a solvent-rinsed Teflon solvent guide to the manifold cover and align with the collection vial.

7.2.9 Add 10 mL of diethyl ether/hexane (10/90, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial.

7.3 Concentrate the eluate using the solvent exchange technique (See Section 7.1). Adjust the final volume according to the analytical method. Analyze by the appropriate GC or GC/MS method.

NOTE: Final extract that contain any methylene chloride or ether will cause damage to the electron capture detector (ECD), so please be certain the solvent exchange is complete!

7.4 Chlorinated Hydrocarbons by GAM 8121

7.4.1 Reduce the sample extract volume to 1 mL prior to cleanup. The extract solvent must be hexane.

7.4.2 Use the cartridge cleanup technique in section 7.3.

7.4.3 Pre-elute the column with 10 mL of petroleum ether. Discard the eluate and, just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract to the column by decantation and subsequent petroleum ether washings. Discard the eluate. Just prior to exposure of the sodium sulfate layer to the air, begin eluting the column with 10 mL of petroleum ether and collect the eluate in a 300 mL TurboVap concentrator tube. This fraction should contain all of the chlorinated hydrocarbons below, for BHC isomers elute the column with 10% ethyl ether in hexane (v/v)and combine with the petroleum ether fraction prior to the concentration step 7.5.4 below:

2-Chloronaphthalene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
1,2,4-Trichlorobenzene.

7.4.4 Concentrate the fraction, using the nitrogen blowdown technique in section 7.1. Adjust the final volume of the cleaned-up extract to whatever volume is required (1-10 mL).

NOTE: Final extract that contain any methylene chloride or ether will cause damage to the electron capture detector (ECD), so please be certain the solvent exchange is complete!

7.5 Nitrosamines by GAM 8270B
(Separation of nitrosodiphenylamine from diphenylamine)

7.5.1 Reduce the sample extract volume to 1 mL prior to cleanup.

7.5.2 Use the cartridge cleanup technique in section 7.3.

7.5.3 Pre-elute the column with 10 mL of ethyl ether/pentane (15:85) (v/v). Discard the eluate and, just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 1 mL sample extract onto the column using an additional 2 mL of pentane to complete the transfer.

7.5.4 Elute the column with 10 mL of ethyl ether/pentane (15:85) (v/v) and discard the eluate. This fraction will contain the diphenylamine, if it is present in the extract. Diphenylamine is not a target compound; however, nitrosodiphenylamine is a target compound that is converted by thermal degradation to diphenylamine in the injection port of the GC. Analyzing the acetone/ethyl ether (5:95)(v/v) fraction collected in section 7.6.5 proves the presence of nitrosodiphenylamine by excluding diphenylamine as an interference.

7.5.5 Next, elute the column at approximately 2 mL/minute with 10 mL of acetone/ethyl ether (5:95)(v/v) into a 300 mL TurboVap concentrator tube. This fraction will contain all of the nitrosamines listed in the scope of the method.

7.5.6 Add 15 mL of methanol to the collected fraction, concentrate using pentane to prewet the K-D column and set the water bath at 70 to 75-C. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of pentane.

7.5.7 Use the Kuderna Danish concentration technique to concentrate the eluate from above. The Turbovap cannot be used for concentration of this fraction because it cannot reach the temperature necessary for this step.

7.6 Organochlorine Pesticides by GAM 8081
(see Tables 1 and 2 for fractionation patterns of compounds tested)

7.6.1 Reduce the sample extract volume to 1 mL prior to cleanup. The extract solvent must be hexane.

7.6.2 Use the chromatographic column method in section 7.2 or the cartridge technique in section 7.3.

7.6.4 Elute the column with 10 mL of 10% ethyl ether in hexane (v/v) using a drip rate of about 2 mL/min.

7.6.5 Concentrate the fraction, using the nitrogen blowdown technique in section 7.1. Adjust the final volume of the cleaned-up extract to whatever volume is required (1-10 mL).

8.0 QUALITY CONTROL

8.1 Refer to GAM 8081, GAM 8121 and GAM 8270B for specific quality control procedures for these respective methods.

8.2 The analyst should demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples.

8.3 For sample extracts that are cleaned up using this method, the associated quality control samples (e.g. - LCS, MS, MSD and prep blank) should also be processed through this cleanup method.

9.0 METHOD PERFORMANCE

9.1 Table 1 indicates the recoveries of chlorinated pesticides and PCB's after elution through a Florisil column.

10.0 REFERENCES

10.1 Gordon, A.J. and R.A. Ford, The Chemist's Companion: A Handbook of Practical Data Techniques, and References (New York: John Wiley & Sons, Inc.), pp. 372 374, and 375, 1972.

10.2 Floridin of ITT System, Florisil: Properties, Application, Bibliography, Pittsburgh, Pennsylvania, 5M381DW.

10.3 Mills, P.A., "Variation of Florisil Activity; Simple Method for Measuring Absorbent Capacity and its use in Standardizing Florisil Columns," Journal of the Association of Official Analytical Chemists, 51, 29, 1968.

10.4 U.S. Food and Drug Association, Pesticides Analytical Manual (Volume 1), July 1985.

10.5 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

10.6 U. S. EPA, "Test Methods for Evaluating Solid Waste" Third Edition Revision 1, September 1994.

TABLE 1: RECOVERIES OF CHLORINATED PESTICIDES AND PCBs

Parameter	Percent Recovery (10% ethyl ether in hexane (v/v))
Aldrin	100
alpha-BHC	100
beta-BHC	97
delta-BHC	98
gamma-BHC	100
Chlordane	100
4,4'-DDD	99
4,4'-DDE	98
4,4'-DDT	100
Dieldrin	100
Endosulfan I	94
Ensosulfan II	91
Endosulfan sulfate	106
Endrin 4	96
Endrin aldehyde	88
Heptachlor	100
Heptachlor epoxide	100
Toxaphene	96
PCB-1016	97
PCB-1221	97
PCB-1232	95
PCB-1242	97
PCB-1248	103
PCB-1254	90
PCB-1260	95

SOURCE: GEO Analytical Inc. replicate LCS analyses.

Attachment QAPP-B15

Sulfur Cleanup
Method 3660A

GCM 3660A
Sulfur Cleanup
Revision 0 : 01/10/97

	CAS #
ANALYTE: Sulfur	7704-34-9

INSTRUMENTATION: N/A

1.0 SCOPE AND APPLICATION

1.1 Elemental sulfur is encountered in many sediment samples (generally specific to different areas in the country), marine algae, and some industrial wastes. The solubility of sulfur in various solvents is very similar to the organochlorine and organophosphorus pesticides. Therefore, the sulfur interference follows along with the pesticides through the normal extraction and cleanup techniques. In general, sulfur will usually elute entirely in Fraction 1 of the Florisil cleanup (GCM 3620).

1.2 Sulfur will be quite evident in gas chromatograms obtained from electron capture detectors used for organochlorine pesticide analysis. If the gas chromatograph is operated at the normal conditions for pesticide analysis, the sulfur interference can completely mask the region from the solvent peak through Aldrin.

1.3 Three techniques for the elimination of sulfur are detailed within this method: (1) the use of copper powder; (2) the use of mercury; and (3) the use of tetrabutylammonium-sulfite. Tetrabutylammonium-sulfite causes the least amount of degradation of a broad range of pesticides and organic compounds, while copper and mercury may degrade organophosphorus and some organochlorine pesticides.

2.0 SUMMARY OF METHOD

2.1 The sample to undergo cleanup is mixed with either copper, mercury, or tetrabutylammonium (TBA)-sulfite. The mixture is shaken and the extract is removed from the sulfur cleanup reagent.

3.0 INTERFERENCES

3.1 Removal of sulfur using copper:

3.1.1 The copper must be very reactive. Therefore, all oxides of copper must be removed so that the copper has a shiny, bright appearance.

3.1.2 The sample extract must be vigorously agitated with the reactive copper for at least one minute.

4.0 APPARATUS AND MATERIALS

4.1 Mechanical shaker or mixer - Vortex Genie or equivalent.

4.2 Pipets, disposable - Pasteur type.

4.3 Centrifuge tubes, calibrated - 12 mL.

4.4 Glass bottles or vials - 20 mL and 40 mL, with Teflon-lined screw caps or crimp tops.

4.5 Kuderna-Danish (K-D) apparatus.

4.5.1 Concentrator tube - 10 mL graduated (Kontes K-570050-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.

4.5.2 Evaporation flask - 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

4.5.3 Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

4.5.4 Snyder column - Two ball micro (Kontes K-569001-0219 or equivalent).

4.5.5 Springs - 1/2 inch (Kontes K-662750 or equivalent).

4.6 Zymark TurboVap - Nitrogen blowdown apparatus.

4.6.1 300 mL concentrator tubes for Turbovap.

4.6.2 6 position racks for Zymark tubes.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water. De-ionized water or tap water must be passed through a bed of activated charcoal to remove any organic contaminants. The use of plastic tubing except for tubing made from PTFE or PFE.

5.3 Nitric acid, HNO3, dilute.

5.4 Solvents

5.4.1 Acetone, CH3COCH3 - Pesticide quality or equivalent.

5.4.2 Hexane, C6H,4 - Pesticide quality or equivalent.

5.4.3 2-Propanol, CH3CH(OH)CH3 - Pesticide quality or equivalent.

5.5 Copper powder - Remove oxides by treating with dilute nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen. (Copper, fine granular Mallinckrodt 4649 or equivalent).

5.6 Mercury, triple distilled.

5.7 Tetrabutylammonium (TBA) sulfite reagent

5.7.1 Tetrabutylammonium hydrogen sulfate, [CH3(CH2)3]4NHSO4.

5.7.2 Sodium sulfite, Na2SO3.

5.7.3 Prepare reagent by dissolving 3.39 g tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. To remove impurities, extract this solution three times with 20 mL portions of hexane. Discard the hexane extracts, and add 25 g sodium sulfite to the water solution. Store the resulting solution, which is saturated with sodium sulfite, in an amber bottle with a Teflon-lined screw cap. This solution can be stored at room temperature for at least one month.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Water and soil samples are stored refrigerated at 4°C. Water samples must be extracted within seven (7) days and soil samples extracted within 14 days of sample collection.

6.2 Extracts must be stored under refrigeration and must be analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Removal of sulfur using copper

7.1.1 Concentrate the sample to exactly 1.0 mL or other known volume. Perform concentration using the TurboVap (nitrogen blowdown technique in GAM 3510B Section 7.10 or GAM 3550A Section 7.3.6)

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

7.1.2 If the sulfur concentration is such that crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable pipet leaving the excess sulfur in the K-D tube. Transfer 1.0 mL of the extract to a calibrated centrifuge tube.

7.1.3 Add approximately 2 g of cleaned copper powder (to the 0.5 mL mark) to the centrifuge tube. Mix for at least 1 min on the mechanical shaker.

7.1.4 Separate the extract from the copper by drawing off the extract with a disposable pipet and transfer to a clean vial. The volume remaining still represents 1.0 mL of extract.

[NOTE: This separation is necessary to prevent further degradation of the pesticides.]

7.2 Removal of sulfur using mercury

[NOTE: Mercury is a highly toxic metal and therefore, must be used with great care. Prior to using mercury, it is recommended that the analyst become acquainted with proper handling and cleanup techniques associated with this metal.]

7.2.1 Concentrate the sample extract to exactly 1.0 mL or other known volume. Perform concentration using the Zymark TurboVap (nitrogen blowdown technique in GAM 3510B Section 7.10 or GAM 3550A Section 7.3.6).

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

7.2.2 Pipet 1.0 mL of the extract into a clean concentrator tube or Teflon-sealed vial.

7.2.3 Add one to three drops of mercury to the vial and seal. Agitate the contents of the vial for 15-30 sec. Prolonged shaking (2 hr) may be required. If so, use a mechanical shaker.

7.2.4 Separate the sample from the mercury by drawing off the extract with a disposable pipet and transfer to a clean vial.

7.3 Removal of sulfur using TBA-sulfite

7.3.1 Concentrate the sample extract to exactly 1.0 mL or other known volume. Perform concentration using the Zymark TurboVap (nitrogen blowdown technique in GAM 3510B Section 7.10 or GAM 3550A Section 7.3.6).

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

7.3.2 Transfer 1.0 mL of the extract to a 50 mL clear glass bottle or vial with a Teflon-lined screw-cap. Rinse the concentrator tube with 1 mL of hexane, adding the rinsings to the 50 mL bottle.

7.3.3 Add 1.0 mL TBA-sulfite reagent and 2 mL 2-propanol, cap the bottle, and shake for at least 1 min. If the sample is colorless or if the initial color is unchanged, and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 0.100 g portions until a solid residue remains after repeated shaking.

7.3.4 Add 5 mL organic free reagent water and shake for at least 1 min. Allow the sample to stand for 5-10 min. Transfer the hexane layer (top) to a concentrator tube and concentrate the extract to approximately 1.0 mL with the micro K-D Technique (Section 7.3.5) or the Nitrogen Blowdown Technique (Section 7.3.6). Record the actual volume of the final extract.

7.3.5 Micro-Snyder Column Technique

7.3.5.1 Add another one or two clean boiling chips to the concentrator tube and attach a two ball micro-Snyder column. Prewet the column by adding about 0.5 mL of hexane to the top of the column. Place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in

the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 5-10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joints with about 0.2 mL of solvent and add to the concentrator tube. Adjust the final volume to approximately 1.0 mL with hexane.

7.3.6 Nitrogen Blowdown Technique

7.3.6.1 Place the concentrator tube in the TurboVap water bath (set at approximately 35-C) and evaporate the solvent volume to 1.0-2.0 mL, using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

7.3.6.2 The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry.

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

7.4 Analyze the cleaned up extracts by gas chromatography.

8.0 QUALITY CONTROL

8.1 LCS, MS, MSD and preparation blanks must be processed using this technique if analytical samples are cleaned up using this method.

8.2 All reagents should be checked prior to use to verify that interferences do not exist. The preparation or reagent blank processed with this procedure will verify that samples are free of interferences.

8.3 Whenever possible, either copper powder or TBA-sulfite will be used instead of mercury cleanup to minimize the potential for mercury contamination in the laboratory and minimize mercury waste disposal problems.

9.0 METHOD PERFORMANCE

9.1 Table 1 indicates the effect of using copper and mercury to remove sulfur on the recovery of certain pesticides.

10.0 REFERENCES

10.1 Loy, E.W., private communication.

10.2 Goerlitz, D.F. and L.M. Law, Bulletin for Environmental Contamination and Toxicology, 6, 9 (1971).

10.3 U.S. EPA Contract Laboratory Program, statement of Work for Organic Analysis, Revision, July 1985.

10.4 U. S. EPA, "Test Methods for Evaluating Solid Waste" Third Edition Revision 1, September 1994.

Table 1.

EFFECT OF MERCURY AND COPPER ON PESTICIDES

Pesticide	Percent Recovery(a) using: Mercury	Copper
Aroclor 1254	97.10	104.26
Lindane	75.73	94.83
Heptachlor	39.84	5.39
Aldrin	95.52	93.29
Heptachlor epoxide	69.13	96.55
DDE	92.07	102.91
DDT	78.78	85.10
BHC	81.22	98.08
Dieldrin	79.11	94.90
Endrin	70.83	89.26

(a) Percent recoveries cited are averages based on duplicate analyses for all compounds other than for Aldrin and BHC. For Aldrin, four and three determinations were averaged to obtain the result for mercury and copper, respectively. Recovery of BHC using copper is based on one analysis.

Attachment QAPP-B16

Sulfuric Acid/Permanganate
Method 3665

GCM 3665

Sulfuric Acid/Permanganate Cleanup
Revision 1.0 : 06/10/99

1.0 SCOPE AND APPLICATION

1.1 This method is suitable for the rigorous cleanup of sample extracts prior to analysis for polychlorinated biphenyls. This method should be used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs. This method cannot be used to cleanup extracts for other target analytes, as it will destroy most organic chemicals including the pesticides Aldrin, Dieldrin, Endrin, Endosulfan(I and II), and Endosulfan sulfate.

2.0 SUMMARY OF METHOD

2.1 An extract is solvent exchanged to hexane, then the hexane is sequentially treated with (1) concentrated sulfuric acid and (2) 5% aqueous potassium permanganate. Appropriate caution must be taken with these corrosive reagents.
2.2 Blanks and replicate analysis samples must be subjected to the same cleanup as the samples associated with them.
2.3 It is important that all the extracts be exchanged to hexane before initiating the following treatments.

3.0 INTERFERENCES

3.1 This technique will not destroy chlorinated benzenes, chlorinated naphthalenes (Halowaxes), and a number of chlorinated pesticides.

4.0 APPARATUS

4.1 Syringe or Class A volumetric pipet, glass; 5.0 mL.
4.2 Vials - 1.8 and 20 mL, glass with Teflon or aluminum foil lined screw caps or crimp tops.
4.3 Concentrator apparatus: Zymark Turbovap with 200 ml tubes.
4.4 Branson 2200 Ultrasonic bath.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in the GEO Analytical Inc. QA/QC manual.
5.3 Sulfuric acid/Water, H_2SO_4/H_2O, (1:1, v/v). EM SX1244TP-1
5.4 Hexane: Pesticide quality, Burdick & Jackson #217-4 or equivalent.

5.5 Potassium permanganate, KMnO4, 5 percent aqueous solution (w/v). Aldrich # 22,346-8 or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Water and soil samples are stored refrigerated at 4°C. Water samples must be extracted within seven (7) days and soil samples extracted within 14 days of sample collection.

6.2 Extracts must be stored under refrigeration and must be analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Sulfuric acid cleanup

7.1.1 Using a syringe or a volumetric pipet, transfer 1.0 mL of the hexane extract to a 9 mL vial and, in a fume hood, carefully add 5 mL of the 1:1 sulfuric acid/water solution.

CAUTION: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

7.1.2 Cap the vial tightly and vortex for one minute. A vortex must be visible in the vial.

CAUTION: Stop the vortexing immediately if the vial leaks, AVOID SKIN CONTACT, SULFURIC ACID BURNS.

7.1.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer; it should not be highly colored nor should it have a visible emulsion or cloudiness.

7.1.4 If a clean phase separation is achieved, proceed to Section 7.1.7.

7.1.5 If the hexane layer is colored or the emulsion persists for several minutes, remove the sulfuric acid layer from the vial and dispose of it properly. Add another 5 mL of the clean 1:1 sulfuric acid/water.

NOTE: Do not remove any hexane at this stage of the procedure.

7.1.6 Vortex the sample for one minute and allow the phases to separate.

7.1.7 Transfer the hexane layer to a clean 9 mL vial.

7.1.8 Add an additional 1 mL of hexane to the sulfuric acid layer, cap and shake. This second extraction is done to ensure quantitative transfer of the PCBs and Toxaphene.

7.1.9 Remove the second hexane layer and combine with the hexane from Section 7.1.7.

7.2 Permanganate cleanup

7.2.1 Add 5 mL of the 5 percent aqueous potassium permanganate solution to the combined hexane fractions from 7.1.9.

CAUTION: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

7.2.2 Cap the vial tightly and vortex for 1 minute. A vortex must be visible in the vial.

CAUTION: Stop the vortexing immediately if the vial leaks. AVOID SKIN CONTACT, POTASSIUM PERMANGANATE BURNS.

7.2.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer, it should not be highly colored nor should it have a visible emulsion or cloudiness.

7.2.4 If a clean phase separation is achieved, proceed to Section 7.2.7.

7.2.5 If the hexane layer is colored or the emulsion persists for several minutes, remove the permanganate solution from the vial via a glass pipette and dispose of it properly. Add another 5 mL of the clean aqueous permanganate solution.

7.2.6 Vortex the sample and allow the phases to separate.

7.2.7 Transfer the hexane layer to a clean 10 mL vial.

7.2.8 Add an additional 1 mL of hexane to the permanganate layer, cap the vial securely and shake. This second extraction is done to ensure quantitative transfer of the PCBs and Toxaphene.
NOTE: Do not remove any hexane at this stage of the procedure.

7.2.9 Remove the second hexane layer and combine with the hexane from Section 7.2.7.

7.3 Final preparation

7.3.1 Reduce the volume of the combined hexane layers to the original volume (1 mL) using the Zymark Turbovap.

7.3.1.1 Place the concentrator tube in a warm water bath (approximately 35-C) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

7.3.1.2 The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample(i.e., the solvent level should be below the level of

the water bath). Under normal operating conditions, the extract should not be allowed to become dry.

7.3.4 Remove any remaining organochlorine pesticides from the extracts using Florisil Column Cleanup (GCM 3620) or Silica Gel Cleanup (GCM 3630).

7.3.5 The extracts obtained may now be analyzed for the target analytes using the appropriate organic technique(s) (see Section 4.3 of this Chapter). If analysis of the extract will not be performed immediately, stopper the concentrator tube and store in a refrigerator. If the extract will be stored longer than 2 days, it should be transferred to a vial with a Teflon lined screw cap or crimp top, and labeled appropriately.

8.0 QUALITY CONTROL

8.1 Refer to GAM 8081 for specific quality control procedures.

9.0 METHOD PERFORMANCE

9.1 No performance data are currently available.

10.0 REFERENCES

10.1 Environmental Protection Agency, November 1986, SW846 Third Edition, "Test Methods For Evaluating Solid Waste", Office of Solid Waste and Emergency Response, Washington, DC 20460.

ATTACHMENT C

GEOANALYTICAL, INC. QUALITY CONTROL CRITERIA

TABLE 1.

QUALITY CONTROL FOR ORGANIC CONTAMINANTS IN WATER METHODS

	CAM 602	CAM 8020	CAM 8081	CAM 610/8100	CAM 8021	CAM 8260	CAM 8270	CAM 418.1	CAM 8015 Modified
Frequency of method blanks	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch
Frequency of Instrument or Reagent blanks	one per ten analytical samples.	one per ten analytical samples.	one per twenty analytical samples.	one per ten analytical samples.	one per ten analytical samples	one per 12 hour clock	one per 12 hour clock	one per ten analytical samples.	one per ten analytical samples.
Surrogates	placed in all samples, blanks, spikes, duplicates. Acceptable recovery ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. Acceptable recovery ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. Acceptable recovery ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. Acceptable recovery ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. Acceptable recovery ranges 20-150.	placed in all samples, blanks, spikes, duplicates. Acceptable recovery ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. Acceptable recovery ranges in Table 11.	n/a	n/a
Laboratory Control Samples	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch
Frequency of MS and MSD's	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.
Accuracy and Precision	compound dependent, see Table 8C.	compound dependent, see Table 8C.	compound dependent, see Table 8F.	compound dependent, see Table 8E.	compound dependent, see Table 19.	compound dependent, see Table 7A.	compound dependent, see Table 7B.	Table 8D.	Table 8D.
Internal standards	no	no	no	no	no	yes	yes	no	no
Initial Calibration	3 point minimum $\leq$ 10% for average RF	5 point minimum $\leq$ 20% for average RF	5 point minimum $\leq$ 20% for average RF	5 point minimum $\leq$ 20% for average RF	5 point minimum $\leq$ 20% for average RF	5 point minimum $\leq$ 30% RSD for average RF of CCC's, <15% RSD for all others	5 point minimum $\leq$ 30% RSD for average RF	5 point minimum $\leq$ 20% RSD for average RF	5 point minimum $\leq$ 20% RSD for average RF
Continuing calibration check	calibration standard run every 10 samples and must be within $\pm$ 15% diff. of average RF	calibration standard run every 10 samples and must be within $\pm$ 15% diff. of average RF	calibration standard run every 10 samples and must be within $\pm$ 15% diff. of average RF	calibration standard run every 10 samples and must be within $\pm$ 15% diff. of average RF	calibration standard run every 10 samples and must be within $\pm$ 15% diff. of average RF	calibration standard run every 12 hours. CCC's must be within $\pm$ 20% drift criteria.	calibration standard run every 12 hours. CCC's must be within $\pm$ 20% drift criteria.	calibration standard run every 10 samples and must be within $\pm$ 15% diff. of average RF	calibration standard run every 10 samples and must be within $\pm$ 15% diff. of average RF
Tuning Parameters	n/a	n/a	n/a	n/a	n/a	BFB to criteria in Table 5, every 12 hours while samples are being run.	DFTPP to criteria in Table 5, every 12 hours while samples are being run.	n/a	n/a
Detection limits	Table 18.	Table 18.	Table 18.	Table 19.	Table 19.	Table 13.	Table 15.	Table 18.	Table 18

TABLE 2.
QUALITY CONTROL FOR ORGANIC CONTAMINANTS IN SOIL METHODS

	GAM 8020	GAM 8081	GAM 8100	GAM 8121	GAM 8260	GAM 8270	GAM 418.1	GAM 8015 modified
Frequency of method blanks	one per every analytical batch or 20 samples.	one per every analytical batch or 20 samples	one per every analytical batch or 20 samples.	one per every analytical batch or 20 samples.	one per every analytical batch or 20 samples.	one per every analytical batch or 20 samples.	one per every analytical batch or 20 samples	one per every analytical batch or 20 samples.
Frequency of Reagent blanks	One per ten analytical samples.	One per twenty analytical samples.	One per ten analytical samples	One per ten analytical samples.	One per 12 hour clock	One per 12 hour clock.	One per ten analytical samples.	One per ten analytical samples.
Surrogates	placed in all samples, blanks, spikes, duplicates. %R ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. %R ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. %R ranges in Table 11.	placed in all samples, blanks, spikes, duplicates. Acceptable recovery range 10 - 150.	placed in all samples, blanks, spikes, duplicates. %R ranges in Table 11.	Placed in all samples, blanks, spikes, duplicates. %R ranges in Table 11.	n/a	n/a
Laboratory Control Samples	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.
Frequency of MS and MSD	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.
Accuracy Precision	compound dependent see Table 8C.	compound dependent see Table 8F.	compound dependent see Table 8E.	compound dependent see Table 19.	compound dependent see Table 8A.	compound dependent see Table 8B.	Table 8D.	Table 8D.
Internal standards	no	no	no	no	yes	yes	no	no
Initial calibration	5 point minimum ≤ 20% RSD for average RF	5 point minimum ≤ 20% RSD for average RF	5 point minimum ≤ 20% RSD for average RF	5 point minimum ≤ 20% RSD for average RF	5 point minimum ≤ 30% RSD for average RF of CCC's, < 15% RSD for all others	5 point minimum ≤ 30% RSD for average RF of CCC	5 point minimum ≤ 20% RSD for average RF	5 point minimum ≤ 20% RSD for average RF
Continuing Calibration Check	calibration standard run every 10 samples and must be within ± 15 % diff. of average RF	calibration standard run every 10 samples and must be within ± 15 % diff. of average RF	calibration standard run every 10 samples and must be within ± 15 % diff. of average RF	calibration standard run every 10 samples and must be within ± 15 % diff. of average RF	calibration standard run every 12 hours and must be within ± 20 % diff. of average RF	calibration standard run every 12 hours and CCC's must be within ± 20 % drift criteria.	calibration standard run every 10 samples and CCC's must be within ± 20 % drift criteria.	calibration standard run every 10 samples and must be within ± 15 % diff. of average RF
Ending Calibration Check	run at the end of each sequence and must be within ± 15 % diff. of average RF	run at the end of each sequence and must be within ± 15 % diff. of average RF	run at the end of each sequence and must be within ± 15 % diff. of average RF	run at the end of each sequence and must be within ± 15 % diff. of average RF	n/a	n/a	run at the end of each sequence and must be within ± 15 % diff. of average RF	run at the end of each sequence and must be within ± 15 % diff. of average RF

TABLE 3.
QUALITY CONTROL FOR METALS IN WATER METHODS

	GAM200/7	GAM 6010	GAM 245.1	GAM/7470/7471
Frequency of method blanks blanks	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch
Initial Calibration	following instrument calibration and must be less than the minimum reporting limit or less than 10% or the sample concentration.	following instrument calibration and must be less than the minimum reporting limit or less than 10% or the sample concentration.	following instrument calibration and must be less than the minimum reporting limit or less than 10% or the sample concentration.	following instrument calibration and must be less than the minimum reporting limit or less than 10% or the sample concentration.
Initial Calibration	following instrument calibration verify calibration with an independently prepared check standard. % R = 95 - 105 %	following instrument calibration verify calibration with the calibration standard. % R = 95 - 105 %	following instrument calibration verify calibration with an independently prepared check standard. % R = 95 - 105 %	following instrument calibration verify calibration with an independently prepared check standard. % R = 90 - 110 %
Laboratory	one LCS per 20 samples or analytical batch	one LCS per 20 samples or analytical batch	one LCS per 20 samples or analytical batch	one LCS per 20 samples or analytical batch
Frequency of MS and MSD's	one MS/MSD per 20 samples or analytical batch	one MS/MSD per 20 samples or analytical batch	one MS/MSD per 20 samples or analytical batch	one MS/MSD per 20 samples or analytical batch
Accuracy and Precision	compound dependent. see Table 19.	compound dependent. see Table 19.	compound dependent. see Table 19.	compound dependent. see Table 19.
Internal Standards	no	no	no	no
Initial Calibration	one level and a blank	one level and a blank	5 levels and a blank $r^2 \geq 0.995$	5 levels and a blank $r^2 \geq 0.995$
Continuing Calibration Blank Verification (CCB)	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration
Continuing Calibration Verification (CCV)	after every ten samples and at the end of each sequence and must be within 90-100% of initial calibration.	after every ten samples and at the end of each sequence and must be within 90-100% of initial calibration.	after every ten samples and at the end of each sequence and must be within 90-100% of initial calibration.	after every ten samples and at the end of each sequence and must be within 90-100% of initial calibration.
Interference Check Samples (ICS)	Run at the beginning and end of each sequence or every 8 hours. ICSA target analytes must be below reporting limit or less than 10% of the sample concentration. ICSAB target analytes must be within $\pm$ 20% of expected.	Run at the beginning and end of each sequence or every 8 hours. ICSA target analytes must be below reporting limit or less than 10% of the sample concentration. ICSAB target analytes must be within $\pm$ 20% of expected.	n/a	n/a
Mass Calibration and Performance Parameters	n/a	n/a	n/a	n/a
Detection Limits	Table 19.	Table 19.	0.2 ug/L based on a 0.1 L sample	0.2 ug/L based on a 0.1 L sample

TABLE 4.
QUALITY CONTROL FOR METALS IN SOIL METHODS

	GAM 6010	GAM 7471 (MERCURY)
Frequency of method blanks	one per every 20 samples or analytical batch.	one per every 20 samples or analytical batch.
Initial Calibration Blank Verification (ICB)	following instrument calibration and must be less than the minimum reporting limit or less than 10% of the sample concentration.	following instrument calibration and must be less than the minimum reporting limit or less than 10% of the sample concentration.
Initial Calibration Verification (ICV)	following instrument calibration verify calibration with an independently prepared check standard. % R = 95 - 105 %	following instrument calibration verify calibration with an independently prepared check standard. % R = 90 - 110%
Laboratory Control Samples	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.
Frequency of MS and MSD's	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.
Accuracy and Precision	compound dependent see Table 20.	compound dependent see Table 20.
Internal standards	no	no
Initial calibration	one level and a blank	5 levels and a blank $r^2 \geq 0.995$
Continuing Calibraion Blank Verification (CCB)	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration.	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration.
Continuing Calibration Verification (CCV)	after every ten samples and at the end of each sequence and must be within 90 - 100 % of initial calibration.	after every ten samples and at the end of each sequence and must be within 80 - 120 % of initial calibration.
Interference Check Samples (ICS)	Run at the beginning and end of each sequence or every 8 hours. ICSA target analytes must be below reporting limit or less than 10% of the sample concentration. ICSAB target analytes must be ± 20 % of expected.	n/a.
Mass Calibration and Performance Parameters	n/a.	n/a.
Detection limits	Table 19	0.1 mg/Kg based on a 1.0 gram sample

TABLE 5.
ION ABUNDANCE TUNING CRITERIA FOR 4-BROMOFLUOROBENZENE (BFB)
Criteria for 624, 8260

m/e	RELATIVE ABUNDANCE CRITERIA
50	15.0 - 40.0 % of mass 95.
75	30.0 - 60.0 % of mass 95.
95	Base peak, 100 % relative abundance.
96	5.0 - 9.0 % of mass 95.
173	Less than 2.0 % of mass 174.
174	Greater than 50.0 % of mass 95.
175	5.0 - 9.0 % of mass 174.
176	> 95.0 % but < 101.0 % of mass 174.
177	5.0 - 9.0 % of mass 176.

Criteria for 524.2

m/e	RELATIVE ABUNDANCE CRITERIA
50	15.0 - 40.0 % of mass 95.
75	30.0 - 80.0 % of mass 95.
95	Base peak, 100 % relative abundance.
96	5.0 - 9.0 % of mass 95.
173	Less than 2.0 % of mass 174.
174	Greater than 50.0 % of mass 95.
175	5.0 - 9.0 % of mass 174.
176	> 95.0 % but < 101.0 % of mass 174.
177	5.0 - 9.0 % of mass 176.

ION ABUNDANCE TUNING CRITERIA FOR DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Criteria used for EPA Method 625

m/e	RELATIVE ABUNDANCE CRITERIA
51	30 - 60 % of mass 198.
68	Less than 2 % of mass 69.
70	Less than 2 % of mass 69.
127	40 - 60 % of mass 198.
197	Less than 1 % of mass 198.
198	Base peak, 100% relative abundance
199	5 - 9 % of mass 198.
275	10 - 30 % of mass 198.
365	Greater than 1 % of base peak.
441	Present but less than mass 443.
442	Greater than 40 % of mass 198.
443	17 - 23 % of mass 442.

Criteria used for EPA Methods 525 and 8270

m/e	RELATIVE ABUNDANCE CRITERIA
51	10 - 80 % of mass 198.
68	Less than 2 % of mass 69.
70	Less than 2 % of mass 69.
127	10 - 80 % of mass 198.
197	Less than 1 % of mass 198.
198	Base peak or >50 % of mass 442
199	5 - 9 % of mass 198.
275	10 - 60 % of mass 198.
365	Greater than 1 % of base peak.
441	Present but less than mass 443
442	Base peak or > 50 % of mass 198
443	15 - 24 % of mass 442.

TABLE 6.
CALIBRATION AND SYSTEM PERFORMANCE CHECK CRITERIA FOR SW846 METHOD 8260

Calibration Check Compounds (CCC)

CCC's for VOLATILE ORGANICS	Recommended %RSD for Linear Fit	Maximum %RSD for Initial Calibration	Maximum %Drift for Continuing Calibration
1,1-Dichloroethene	15	30	20
Chloroform	15	30	20
1,2-Dichloropropane	15	30	20
Toluene	15	30	20
Ethylbenzene	15	30	20
Vinyl Chloride	15	30	20

System Performance Check Compounds (SPCC)

SPCC's for VOLATILE ORGANICS	Minimum RRF	Minimum RRF for Continuing Calibration
Chloromethane	0.10	0.10
1,1-Dichloroethane	0.10	0.10
Bromoform	> 0.10	> 0.10
Chlorobenzene	0.30	0.30
1,1,2,2-Tetrachloroethane	0.30	0.30

* - % Drift = $(C_1 - C_C)/C_1$ X 100 where C_1 = concentration of the CCC standard
C_C = measured concentration using selected quantitation method.

Initial calibration criteria for the remaining compounds states that if %RSD for the five point curve is less than or equal to 15%, the relative response factor is assumed to be constant over the calibration range and the average response factor may be used. If the %RSD is greater than 15%, a calibration curve is constructed using the area ratio (A/A_{is}) versus concentration using the first or higher order regression fit of the five point curve. The regression fit that introduces the least amount of error should be selected.

TABLE 7.
Acceptance Limits for Laboratory Control Samples, Matrix Spikes and Spike Duplicates

(A) VOLATILE ORGANICS BY GC/MS

COMPOUNDS	Water Methods (EPA, SW846)	LCS Accuracy %R	Water Accuracy %R	Water Precision RPD	Soil Methods (SW846)	Soil Accuracy %R	Soil Precision RPD
1,1-Dichloroethene	624, 8240, 8260	70 - 125	73 - 140	16	8240, 8260	36 - 156	19
Trichloroethene	624, 8240, 8260	75 - 123	65 - 137	11	8240, 8260	50 - 119	21
Benzene	624, 8240, 8260	80 - 116	76 - 133	11	8240, 8260	55 - 115	21
Toluene	624, 8240, 8260	76 - 120	62 - 147	13	8240, 8260	54 - 117	19
Chlorobenzene	624, 8240, 8260	85 - 115	68 - 143	12	8240, 8260	51 - 124	21

(B) SEMI-VOLATILE ORGANICS BY GC/MS

COMPOUNDS	Water Methods (EPA, SW846)	Water Accuracy %R	Water Precision RPD	Soil Methods (SW846)	Soil Accuracy %R	Soil Precision RPD
Phenol	625, 8270	12 - 112	42	8270	47 - 114	33
2-Chlorophenol	625, 8270	27 - 123	40	8270	49 - 115	24
4-Chloro-3-methylphenol	625, 8270	30 - 120	31	8270	39 - 132	23
4-Nitrophenol	625, 8270	10 - 80	50	8270	11 - 114	50
Pentachlorophenol	625, 8270	32 - 129	27	8270	5 - 142	17
1,4-Dichlorobenzene	625, 8270	36 - 97	28	8270	29 - 133	26
N-Nitroso-di-n-propylamine	625, 8270	23 - 121	38	8270	48 - 129	23
1,2,4-Trichlorobenzene	625, 8270	39 - 98	28	8270	44 - 121	23
Acenaphthene	625, 8270	46 - 118	31	8270	56 - 125	19
2,4-Dinitrotoluene	625, 8270	30 - 121	41	8270	46 - 114	21
Pyrene	625, 8270	50 - 129	37	8270	50 - 140	24

(C) AROMATIC VOLATILE ORGANICS BY GC (LOW LEVEL METHOD)

COMPOUNDS	Water Methods (EPA, SW846)	Water Accuracy %R	Water Precision RPD	Soil Methods (SW846)	Soil Accuracy %R	Soil Precision RPD
Benzene	602, 8020	82 - 115	0 - 6	8020	79 - 115	0 - 6
Toluene	602, 8020	83 - 116	0 - 6	8020	78 - 117	0 - 7
Chlorobenzene	602, 8020	86 - 116	0 - 4	8020	79 - 115	0 - 8
Ethylbenzene	602, 8020	85 - 115	0 - 5	8020	74 - 121	0 - 7
m&p-Xylene	602, 8020	84 - 115	0 - 7	8020	72 - 123	0 - 7
o-Xylene	602, 8020	83 - 117	0 - 6	8020	75 - 122	0 - 6
1,3-Dichlorobenzene	602, 8020	88 - 116	0 - 7	8020	62 - 127	0 - 8
1,4-Dichlorobenzene	602, 8020	85 - 112	0 - 7	8020	66 - 127	0 - 8
1,2-Dichlorobenzene	602, 8020	87 - 116	0 - 5	8020	63 - 125	0 - 7

(D) TOTAL PETROLEUM HYDROCARBONS

COMPOUNDS	Water Methods (EPA, SW846)	Water Accuracy %R	Water Precision RPD	Soil Methods (SW846)	Soil Accuracy %R	Soil Precision RPD
Total petroleum hydrocarbons IR	418.1	81 - 112	0 - 6	9073	82 - 102	0 - 8
TPH GRO GC	8015	71 - 115	0 - 17	8015	85 - 120	0 - 20
TPH DRO GC	8015	48 - 121	0 - 15	8015	84 - 122	0 - 18

TABLE 7. (cont.)
Acceptance Limits for Laboratory Control Samples, Matrix Spikes and Spike Duplicates

(E) POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHIC METHODS 610 and 8100

COMPOUNDS	Water Methods (EPA, SW846)	Water Accuracy %R	Water Precision RPD	Soil Methods (SW846)	Soil Accuracy %R	Soil Precision RPD
Naphthalene	610, 8100	56 - 108	0 - 15	8100	34 - 138	0 - 25
Acenaphthylene	610, 8100	65 - 115	0 - 11	8100	43 - 134	0 - 26
Acenaphthene	610, 8100	68 - 114	0 - 10	8100	48 - 133	0 - 25
Fluorene	610, 8100	73 - 117	0 - 11	8100	44 - 142	0 - 24
Phenanthrene	610, 8100	74 - 117	0 - 14	8100	49 - 141	0 - 26
Anthracene	610, 8100	75 - 122	0 - 17	8100	47 - 145	0 - 28
Fluoranthene	610, 8100	79 - 117	0 - 13	8100	51 - 149	0 - 22
Pyrene	610, 8100	77 - 119	0 - 12	8100	50 - 148	0 - 22
Benzo(a)anthracene	610, 8100	79 - 117	0 - 10	8100	49 - 151	0 - 29
Chrysene	610, 8100	74 - 123	0 - 9	8100	50 - 152	0 - 29
Benzo(b)fluoranthene	610, 8100	72 - 119	0 - 15	8100	45 - 157	0 - 30
Benzo(k)fluoranthene	610, 8100	72 - 125	0 - 17	8100	51 - 144	0 - 31
Benzo(a)pyrene	610, 8100	74 - 120	0 - 12	8100	49 - 149	0 - 30
Indeno(1,,2,3-cd)pyrene	610 8100	65 - 128	0 - 11	8100	50 - 153	0 - 30
Dibenzo(a,h)anthracene	610, 8100	69 - 126	0 - 14	8100	46 - 153	0 - 30
Benzo(ghi)perylene	610, 8100	65 - 126	0 - 16	8100	54 - 142	0 - 21

(F) ORGANOCHLORINE PESTICIDES and POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHIC METHODS 608 and 8081

COMPOUNDS	Water Methods (EPA/SW846)	Water Accuracy %R	Water Precision RPD	Soil Methods (SW846)	Soil Accuracy %R	Soil Precision RPD
Aldrin	608/8081	40 - 120	0 - 20	8081	34 - 132	0 - 43
gamma-BHC (Lindane)	608/8081	56 - 123	0 - 15	8081	46 - 127	0 - 50
4,4'-DDT	608/8081	38 - 127	0 - 27	8081	23 - 134	0 - 50
Dieldrin	608/8081	52 - 126	0 - 18	8081	31 - 134	0 - 38
Endrin	608/8081	56 - 121	0 - 21	8081	42 - 139	0 - 45
Heptachlor	608/8081	40 - 131	0 - 20	8081	35 - 130	0 - 31
Aroclor 1016	608/8081	50 - 150	0 - 50	8081	50 - 150	0 - 50
Aroclor 1221	608/8081	50 - 150	0 - 50	8081	50 - 150	0 - 50
Aroclor 1232	608/8081	50 - 150	0 - 50	8081	50 - 150	0 - 50
Aroclor 1242	608/8081	50 - 150	0 - 50	8081	50 - 150	0 - 50
Aroclor 1248	608/8081	50 - 150	0 - 50	8081	50 - 150	0 - 50
Aroclor 1254	608/8081	50 - 150	0 - 50	8081	50 - 150	0 - 50
Aroclor 1260	608/8081	50 - 150	0 - 50	8081	50 - 150	0 - 50

TABLE 8.
CALIBRATION AND SYSTEM PERFORMANCE CHECK CRITERIA FOR SW846 METHOD 8270

Calibration Check Compounds (CCC)

SEMI-VOLATILES	Recommended %RSD for Initial Calibration	Maximum %RSD for Initial Calibration	Maximum % Drift for Continuing Calibration Check
Phenol	15 %	30 %	20 %
1,4-Dichlorobenzene	15 %	30 %	20 %
2-Nitrophenol	15 %	30 %	20 %
2,4-Dichlorophenol	15 %	30 %	20 %
Hexachlorobutadiene	15 %	30 %	20 %
4-Chloro-3-methylphenol	15 %	30 %	20 %
2,4,6-Trichlorophenol	15 %	30 %	20 %
Acenaphthene	15 %	30 %	20 %
N-Nitrosodiphenylamine	15 %	30 %	20 %
Pentachlorophenol	15 %	30 %	20 %
Fluoranthene	15 %	30 %	20 %
Di-n-octylphthalate	15 %	30 %	20 %
Benzo (a) pyrene	15 %	30 %	20 %

System Performance Check Compounds (SPCC)

SEMI-VOLATILES	Minimum Response Factor for Initial Calibration	Minimum Response Factor for Continuing Calibration
N-Nitroso-di-propylamine	0.05	0.05
Hexachlorocyclopentadiene	0.05	0.05
2,4-Dinitrophenol	0.05	0.05
4-Nitrophenol	0.05	0.05

TABLE 9.
DATA REPORTS AND SIGNIFICANT FIGURES

Data reporting follows a general rule of three significant figures when possible. Each method and instrument; however, has a lower limit of measurement which is often less than three significant figures. The table below illustrates how significant figures for each method are determined and reported. Each "#" sign represents a significant figure and each zero is a place holder or non-significant figure. When rounding numerical data to the appropriate number of significant figures the following rules apply :

1. When the number next beyond the last place to be retained is less than 5, the number in the place retained is left unchanged. *Example : 12.34 rounds to three significant figures as 12.3.*

2. When the number next beyond the last place to be retained is greater than 5, the number in the place retained is rounded to the next higher number. *Example : 12.36 rounds to three significant figures as 12.4.*

3. When the number next beyond the last place to be retained is 5, the number in the place retained is rounded to the nearest even number. *Example : 12.35 rounds to three significant figures as 12.4 and 12.45 rounds to 12.4.*

METHOD	Units	Range 1.0 - 10.0	Range 10.0 - 100	Range 100 - 1,000	Range 1,000 - 10,000	Range 10,000 - 100,000
EPA 602, SW846 8020	ug/L	#.#	##.#	###	###0	###00
SW846 8020	ug/Kg	#.#	##.#	###	###0	###00
SW846 8015	ug/L	#.#	##.#	###	###0	###00
SW846 8015	mg/Kg	#.#	##.#	###	###0	###00
EPA 418.1	mg/L	#.#	##.#	###	###0	###00
EPA 418.1	mg/Kg	#.#	##.#	###	###0	###00
EPA 624, SW846 8240	ug/L	#.#	##.#	###	###0	###00
EPA 524.2	ug/L	#.#	##.#	###	###0	###00
SW846 8240, 8260	ug/Kg	#.#	##.#	###	###0	###00
EPA 610	ug/L	#.#	##.#	###	###0	###00
EPA 625, 8270	ug/L	#.#	##.#	###	###0	###00
EPA 200.7, SW846 6010	mg/L	#.#	##.#	###	###0	###00
EPA 200.8, SW846 6020	ug/L	# #	## #	###	###0	###00
SW846 6020	mg/Kg	# #	## #	###	###0	###00
EPA 245.1, SW846 7470	mg/L	# #	# #	###	###	###00
EPA 245.5, SW846 7471	mg/Kg	#.#	##.#	###	###0	###00

METHOD	Units	Range 0.01 - 0.10	Range 0.1 - 10.0	Range 10.0 - 100	Range 100 - 1,000	Range 1,000 - 10,000
SW846 8100	ug/L	0.0#	#.#	#.##	###	###0
SW846 8270	mg/Kg	0.0#	#.#	#.##	###	###0
SW846 6010 (TCLP)	mg/L	0.0#	#.#	#.##	###	###0

TABLE 10.
SURROGATE RECOVERIES FOR GC METHODS

METHOD 602 Surrogates	Acceptable Range for Water
Fluorobenzene	73 - 122
Trifluorotoluene	72 - 123

METHOD 608 Surrogates	Acceptable Range for Water
2,3,7,8-Tetrachloro-o-xylene	39 - 123
Decachlorobiphenyl	7 - 156

METHOD 8020 Surrogates	Acceptable Range for Water	Acceptable Range for Soil	Acceptable Range for High Level Soil
Fluorobenzene	75 - 117	51 - 126	74 - 124
Trifluorotoluene	67 - 125	36 - 133	79 - 117

METHOD 8080/8081 Surrogates	Acceptable Range for Water	Acceptable Range for Soil
2,3,7,8-Tetrachloro-o-xylene	39 - 123	25 - 143
Decachlorobiphenyl	7 - 156	22 - 103

METHOD 8100 Surrogates	Acceptable Range for Water	Acceptable Range for Soil
2-Fluorobiphenyl	66 - 118	70 - 105
ortho-Terphenyl	72 - 122	72 - 114

* Indicates an interim limit.

METHOD 8121 Surrogates	Acceptable Range for Water*	Acceptable Range for Soil*
1,4-Dichloronaphthalene	20 - 150	10 - 150

* Indicates an interim limit.

TABLE 11.
SURROGATE RECOVERIES FOR GC/MS METHODS

METHOD 624 Surrogates	Acceptable Range
1,2-Dichloroethane d4	84 - 123
Toluene d8	91 - 117
4-Bromofluorobenzene	85 - 111
Dibromofluoromethane	88 - 110

METHOD 625 Surrogates	Acceptable Range
2-Fluorophenol	21 - 110
Phenol d5	10 - 110
2,4,6-Tribromophenol	10 - 123
Nitrobenzene d5	35 - 114
2-Fluorobiphenyl	43 - 116
Terphenyl d14	33 - 141

METHOD 8240/8260 Surrogates	Acceptable Range for Water	Acceptable Range for Soil	Acceptable Range for High Level Soil
1,2-Dichloroethane d4	84 - 123	91 - 126	59 - 141
Toluene d8	91 - 117	66 - 136	56 - 152
4-Bromofluorobenzene	85 - 111	67 - 123	48 - 152
Dibromofluoromethane	88 - 110	95 - 104	82 - 118

METHOD 8270 Surrogates	Acceptable Range for Water	Acceptable Range for Soil
2-Fluorophenol	21 - 110	24 - 112
Phenol d5	10 - 110	25 - 121
2,4,6-Tribromophenol	10 - 123	19 - 122
2-Chlorophenol d4	33 - 110*	20 - 130*
1,2 - Dichlorobenzene d4	16 - 110*	20 - 130*
Nitrobenzene d5	35 - 114	23 - 120
2-Fluorobiphenyl	43 - 116	30 - 115
Terphenyl d14	33 - 141	18 -137

* Advisory limits

TABLE 12.
EPA METHOD 624
QC Acceptance Criteria and Detection Limit Summary

COMPOUNDS	Accuracy %R	Precision RPD	Completeness %	MRL (ug/L)	MDL (ug/L)
Chloromethane	D - 273	0 - 20	95	5.0	0.24
Vinyl chloride	D - 251	0 - 20	95	5.0	0.28
Bromomethane	D - 242	0 - 20	95	5.0	0.35
Chloroethane	14 - 230	0 - 20	95	5.0	0.24
Trichlorofluoromethane	17 - 181	0 - 20	95	5.0	0.19
1,1-Dichloroethene	D - 234	0 - 20	95	5.0	0.18
Methylene chloride	D - 221	0 - 20	95	5.0	0.41
trans-1,2-Dichloroethene	54 - 156	0 - 20	95	5.0	0.28
1,1-Dichloroethane	59 - 155	0 - 20	95	5.0	0.22
Chloroform	51 - 138	0 - 20	95	5.0	0.26
1,1,1-Trichloroethane	52 - 162	0 - 20	95	5.0	0.19
Carbon tetrachloride	70 - 140	0 - 20	95	5.0	0.12
Benzene	37 - 151	0 - 20	95	5.0	0.19
1,2-Dichloroethane	49 - 155	0 - 20	95	5.0	0.24
Trichloroethene	71 - 157	0 - 20	95	5.0	0.17
1,2-Dichloropropane	D - 210	0 - 20	95	5.0	0.18
Bromodichloromethane	35 - 155	0 - 20	95	5.0	0.17
2-Chloroethylvinyl ether	D - 305	0 - 20	95	5.0	0.10
cis-1,3-Dichloropropene	D - 227	0 - 20	95	5.0	0.24
Toluene	47 - 150	0 - 20	95	5.0	0.17
trans-1,3-Dichloropropene	17 - 183	0 - 20	95	5.0	0.28
1,1,2-Trichloroethane	52 - 150	0 - 20	95	5.0	0.16
Tetrachloroethene	64 - 148	0 - 20	95	5.0	0.18
Dibromochloromethane	53 - 149	0 - 20	95	5.0	0.12
Chlorobenzene	37 - 160	0 - 20	95	5.0	0.24
Ethylbenzene	37-162	0 - 20	95	5.0	0.24
Bromoform	45 - 169	0 - 20	95	5.0	0.17
1,1,2,2-Tetrachloroethane	46 - 157	0 - 20	95	5.0	0.23
1,3-Dichlorobenzene	59 - 156	0 - 20	95	5.0	0.62
1,4-Dichlorobenzene	18 - 190	0 - 20	95	5.0	0.45
1,2-Dichlorobenzene	18 - 190	0 - 20	95	5.0	0.37
Xylenes (total)	40 - 160	0 - 20	95	5.0	0.17

nd - not determined

TABLE 13.
EPA METHOD SW846 8260
QC Acceptance Criteria and Detection Limit Summary

COMPOUND	Accuracy	Precision	Completeness	Soil MRL	Water MRL	Soil MDL	Water MDL
DICHLORODIFLUOROMETHANE	40 -160	0 - 30	95	5.0	5.0	0.87	0.19
CHLOROMETHANE	D - 273	0 - 30	95	5.0	5.0	0.68	0.24
VINYL CHLORIDE	D- 251	0 - 30	95	5.0	5.0	0.53	0.28
BROMOMETHANE	D - 242	0 - 30	95	5.0	5.0	0.33	0.35
CHLOROETHANE	14 - 230	0 - 30	95	5.0	5.0	0.47	0.24
TRICHLOROFLUOROMETHANE	17 - 181	0 - 30	95	5.0	5.0	0.87	0.19
1,1-DICHLOROETHENE	D - 234	0 - 30	95	5.0	5.0	0.64	0.18
ACETONE	40 - 160	0 - 30	95	25.0	25.0	3.06	0.75
CARBON DISULFIDE	40 - 160	0 - 30	95	5.0	5.0	0.65	0.20
METHYLENE CHLORIDE	D - 221	0 - 30	95	5.0	5.0	0.31	0.41
TRANS-1,2-DICHLOROETHENE	54 - 156	0 - 30	95	5.0	5.0	0.54	0.28
1,1-DICHLOROETHANE	59 - 155	0 - 30	95	5.0	5.0	0.31	0.22
VINYL ACETATE	40 - 160	0 - 30	95	25.0	25.0	0.53	0.19
CIS-1,2-DICHLOROTHENE	40 - 160	0 - 30	95	5.0	5.0	0.44	0.31
2-BUTANONE	40 - 160	0 - 30	95	25.0	25.0	1.27	0.95
CHLOROFORM	51 - 138	0 - 30	95	5.0	5.0	0.69	0.26
1,1,1-TRICHLOROETHANE	52 - 162	0 - 30	95	5.0	5.0	0.67	0.19
CARBON TETRACHLORIDE	70 - 140	0 - 30	95	5.0	5.0	0.76	0.12
BENZENE	37 - 151	0 - 30	95	5.0	5.0	0.43	0.19
1,2-DICHLOROETHANE	49 - 155	0 - 30	95	5.0	5.0	0.37	0.24
TRICHLOROETHENE	71 - 157	0 - 30	95	5.0	5.0	0.66	0.17
1,2-DICHLOROPROPANE	D - 210	0 - 30	95	5.0	5.0	0.48	0.18
BROMODICHLOROMETHANE	35 - 155	0 - 30	95	5.0	5.0	0.38	0.17
CIS-1,3-DICHLOROPROPENE	D - 227	0 - 30	95	5.0	5.0	0.64	0.24
4-METHYL-2-PENTANONE	40 - 160	0 - 30	95	25.0	25.0	2.36	0.24
TOLUENE	47 - 150	0 - 30	95	5.0	5.0	0.51	0.17
TRANS-1,3-DICHLOROPROPENE	17 - 183	0 - 30	95	5.0	5.0	0.61	0.28
1,1,2-TRICHLOROETHANE	52 - 150	0 - 30	95	5.0	5.0	0.46	0.16
TETRACHLOROETHENE	64 - 148	0 - 30	95	5.0	5.0	0.66	0.18
2-HEXANONE	40 - 160	0 - 30	95	25.0	25.0	4.91	0.37
DIBROMOCHLOROMETHANE	53 - 149	0 - 30	95	5.0	5.0	0.58	0.12
CHLOROBENZENE	37 - 160	0 - 30	95	5.0	5.0	0.74	0.24
ETHYLBENZENE	37 - 162	0 - 30	95	5.0	5.0	0.86	0.24
M&P-XYLENE	40 - 160	0 - 30	95	5.0	5.0	0.64	0.34
O-XYLENE	40 - 160	0 - 30	95	5.0	5.0	0.84	0.17
STYRENE	54 - 148	0 - 30	95	5.0	5.0	0.96	0.21
BROMOFORM	45 - 169	0 - 30	95	5.0	5.0	0.86	0.17
1,1,2,2-TETRACHLOROETHANE	46 - 157	0 - 30	95	5.0	5.0	0.76	0.23
2-CHLOROETHYL VINYL ETHER	40 - 160	0 - 30	95	5.0	5.0	0.99	0.10
1,3-DICHLOROBENZENE	59 - 156	0 - 30	95	5.0	5.0	0.73	0.62
1,4-DICHLOROBENZENE	18 - 190	0 - 30	95	5.0	5.0	0.77	0.45
1,2-DICHLOROBENZENE	18 - 190	0 - 30	95	5.0	5.0	0.83	0.37
2,2-DICHLOROPROPANE	40 - 160	0 - 30	95	5.0	5.0	0.70	0.47
BROMOCHLOROMETHANE	17 - 121	0 - 30	95	5.0	5.0	0.24	0.14
DIBROMOMETHANE	D - 152	0 - 30	95	5.0	5.0	0.48	0.62
1,3-DICHLOROPROPANE	D - 123	0 - 30	95	5.0	5.0	0.42	0.36
1,2-DIBROMOETHANE	40 - 160	0 - 30	95	5.0	5.0	0.66	0.80
1,1,1,2-TETRACHLOROETHANE	40 - 160	0 - 30	95	5.0	5.0	0.61	0.37
ISOPROPYLBENZENE	32 - 136	0 - 30	95	5.0	5.0	0.90	0.56
1,2,3-TRICHLOROPROPANE	40 - 160	0 - 30	95	5.0	5.0	0.83	0.65
n-PROPYLBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.43	0.66
BROMOBENZENE	D - 145	0 - 30	95	5.0	5.0	0.52	0.35
1,3,5-TRIMETHYLBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.44	0.48
2-CHLOROTOLUENE	40 - 160	0 - 30	95	5.0	5.0	0.55	0.51
4-CHLOROTOLUENE	40 - 160	0 - 30	95	5.0	5.0	0.56	0.53
t-BUTYLBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.45	0.60
1,2,4-TRIMETHYLBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.60	0.42
sec-BUTYLBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.45	0.55
4-ISOPROPYLTOLUENE	40 - 160	0 - 30	95	5.0	5.0	0.50	0.55
n-BUTYLBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.48	0.62
1,2-DIBROMO-3-CHLOROPROPANE	40 - 160	0 - 30	95	5.0	5.0	0.49	0.55
1,2,4-TRICHLOROBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.74	0.53
HEXACHLOROBUTADIENE	40 - 160	0 - 30	95	5.0	5.0	0.52	0.31
NAPHTHALENE	40 - 160	0 - 30	95	5.0	5.0	0.32	0.89
1,2,3-TRICHLOROBENZENE	40 - 160	0 - 30	95	5.0	5.0	0.99	0.80
1,1-DICHLOROPROPENE	40 - 160	0 - 30	95	5.0	5.0	0.50	0.50
METHYL-t-BUTYL ETHER	40 - 160	0 - 30	95	5.0	5.0	0.78	0.25
n-HEXANE	40 - 160	0 - 30	95	5.0	5.0	0.69	0.95

TABLE 14.
EPA METHOD 625
QC Acceptance Criteria and Detection Limit Summary

COMPOUNDS	Accuracy %R	Precision RPD	Completeness %	MRL (ug/L)	MDL (ug/L)
N-Nitrosodimethylamine	10 - 150	0 - 40	95	25.0	3.9
Phenol	5 - 112	0 - 40	95	5.0	3.9
2-Chlorophenol	23 - 134	0 - 40	95	5.0	3.9
bis(2-Chloroethyl)ether	12 - 158	0 - 40	95	5.0	3.1
1,3-Dichlorobenzene	D - 172	0 - 40	95	5.0	3.7
1,4-Dichlorobenzene	20 - 124	0 - 40	95	5.0	3.6
1,2-Dichlorobenzene	32 - 129	0 - 40	95	5.0	2.9
bis(2-Chloroisopropyl)ether	36 - 166	0 - 40	95	5.0	4.4
Hexachloroethane	40 - 112	0 - 40	95	5.0	4.6
N-Nitroso-di-n-propylamine	D - 230	0 - 40	95	25.0	4.5
Nitrobenzene	35 - 180	0 - 40	95	5.0	4.7
Isophorone	21 - 196	0 - 40	95	5.0	4.8
2-Nitrophenol	29 - 182	0 - 40	95	5.0	4.6
2,4-Dimethylphenol	32 - 119	0 - 40	95	5.0	3.8
bis(2-Chloroethoxy)methane	33 - 184	0 - 40	95	5.0	4.3
2,4-Dichlorophenol	39 - 135	0 - 40	95	5.0	4.7
1,2,4-Trichlorobenzene	44 - 142	0 - 40	95	5.0	3.4
Naphthalene	21 - 133	0 - 40	95	5.0	3.6
Hexachlorobutadiene	24 - 116	0 - 40	95	5.0	3.9
4-Chloro-3-methylphenol	22 - 147	0 - 40	95	5.0	3.6
Hexachlorocyclopentadiene	10 - 150	0 - 40	95	5.0	4.9
2,4,6-Trichlorophenol	37 - 144	0 - 40	95	5.0	4.4
2-Chloronaphthalene	60 - 118	0 - 40	95	5.0	4.6
Acenaphthylene	33 - 145	0 - 40	95	5.0	4.4
Dimethyl phthalate	D - 112	0 - 40	95	5.0	2.7
2,6-Dinitrotoluene	50 - 158	0 - 40	95	5.0	4.2
Acenaphthene	47 - 145	0 - 40	95	5.0	4.1
2,4-Dinitrophenol	D - 191	0 - 40	95	25.0	4.4
4-Nitrophenol	D - 132	0 - 40	95	5.0	2.7
2,4-Dinitrotoluene	39 - 139	0 - 40	95	5.0	2.8
Diethyl phthalate	D - 114	0 - 40	95	5.0	3.9
Fluorene	59 - 121	0 - 40	95	5.0	3.1
4-Chlorophenylphenyl ether	25 - 158	0 - 40	95	5.0	3.1
2-Methyl-4,6-dinitrophenol	D - 181	0 - 40	95	25.0	3.5
N-Nitrosodiphenylamine	10 - 150	0 - 40	95	5.0	1.4
Azobenzene	10 - 150	0 - 40	95	5.0	33.5
4-Bromphenylphenyl ether	53 - 127	0 - 40	95	5.0	2.2
Hexachlorobenzene	D - 152	0 - 40	95	5.0	1.8
Pentachlorophenol	14 - 176	0 - 40	95	5.0	1.8
Phenanthrene	54 - 120	0 - 40	95	5.0	2.3
Anthracene	27 - 133	0 - 40	95	5.0	2.5
Di-n-butyl phthalate	1 - 118	0 - 40	95	5.0	1.9
Fluoranthene	26 - 137	0 - 40	95	5.0	1.2
Benzidine	10 - 150	0 - 40	95	25.0	6.2
Pyrene	52 - 115	0 - 40	95	5.0	0.7
Butyl benzyl phthalate	D - 152	0 - 40	95	5.0	3.4
Benzo(a)anthracene	33 - 143	0 - 40	95	5.0	4.3
3,3'-Dichlorobenzidine	D - 262	0 - 40	95	25.0	2.7
Chrysene	17 - 168	0 - 40	95	5.0	1.2
bis(2-Ethylhexyl) phthalate	8 - 158	0 - 40	95	5.0	2.5
Di-n-octyl phthalate	4 - 146	0 - 40	95	5.0	3.7
Benzo(b)fluoranthene	24 - 159	0 - 40	95	5.0	1.6
Benzo(k)fluoranthene	11 - 162	0 - 40	95	5.0	2.7
Benzo(a)pyrene	17 - 163	0 - 40	95	5.0	1.1
Indeno(1,2,3-cd)pyrene	D - 171	0 - 40	95	5.0	1.4
Dibenzo(a,h)anthracene	D - 227	0 - 40	95	5.0	0.8
Benzo(g,h,i)perylene	D - 219	0 - 40	95	5.0	0.6

nd- not determined

TABLE 15.
EPA METHOD 8270
QC Acceptance Criteria and Detection Limit Summary

COMPOUNDS	Accuracy %R	Precision RPD	Completeness %	Water MRL (ug/L)	Soil MRL (mg/Kg)	Water MDL (ug/L)	Soil MDL (ug/Kg)
N-Nitrosodimethylamine	10 - 150	0 - 40	95	25.0	1.65	3.9	0.05
Phenol	5 - 112	0 - 40	95	5.0	0.330	3.9	0.05
2-Chlorophenol	23 - 134	0 - 40	95	5.0	0.330	3.9	0.07
bis(2-Chloroethyl)ether	12 - 158	0 - 40	95	5.0	0.330	3.1	0.07
1,3-Dichlorobenzene	D - 172	0 - 40	95	5.0	0.330	3.7	0.09
1,4-Dichlorobenzene	20 - 124	0 - 40	95	5.0	0.330	3.6	0.09
1,2-Dichlorobenzene	32 - 129	0 - 40	95	5.0	0.330	2.9	0.07
Benzyl alcohol	10 - 150	0 - 40	95	5.0	0.330	3.9	0.08
2-Methylphenol	10 - 150	0 - 40	95	5.0	0.330	4.9	0.06
bis(2-Chloroisopropyl)ether	36 - 166	0 - 40	95	5.0	0.330	4.4	0.06
4-Methylphenol	10 - 150	0 - 40	95	5.0	0.330	4.9	0.05
Hexachloroethane	40 - 112	0 - 40	95	5.0	0.330	4.6	0.07
N-Nitroso-di-n-propylamine	D - 230	0 - 40	95	25.0	1.65	4.5	0.10
Nitrobenzene	35 - 180	0 - 40	95	5.0	0.330	4.7	0.05
Isophorone	21 - 196	0 - 40	95	5.0	0.330	4.8	0.08
2-Nitrophenol	29 - 182	0 - 40	95	5.0	0.330	4.6	0.10
2,4-Dimethylphenol	32 - 119	0 - 40	95	5.0	0.330	3.8	0.06
bis(2-Chloroethoxy)methane	33 - 184	0 - 40	95	5.0	0.330	4.3	0.09
2,4-Dichlorophenol	39 - 135	0 - 40	95	5.0	0.330	4.7	0.06
1,2,4-Trichlorobenzene	44 - 142	0 - 40	95	5.0	0.330	3.4	0.05
Naphthalene	21 - 133	0 - 40	95	5.0	0.330	3.6	0.07
4-Chloroanaline	10 - 150	0 - 49	95	5.0	0.330	3.9	0.10
Hexachlorobutadiene	24 - 116	0 - 40	95	5.0	0.330	3.9	0.08
4-Chloro-3-methylphenol	22 - 147	0 - 40	95	5.0	0.330	3.6	0.07
2-Methylnaphthalene	10 - 150	0 - 40	95	5.0	0.330	4.0	0.04
Hexachlorocyclopentadiene	10 - 150	0 - 40	95	5.0	0.330	4.9	0.04
2,4,5-Trichlorophenol	10 - 150	0 - 40	95	5.0	0.330	3.4	0.08
2,4,6-Trichlorophenol	37 - 144	0 - 40	95	5.0	0.330	4.4	0.09
2-Chloronaphthalene	60 - 118	0 - 40	95	5.0	0.330	4.6	0.06
2-Nitroanaline	10 - 150	0 - 40	95	5.0	0.330	3.1	0.05
Acenaphthylene	33 - 145	0 - 40	95	5.0	0.330	4.4	0.05
Dimethyl phthalate	D - 112	0 - 40	95	5.0	0.330	2.7	0.05
2,6-Dinitrotoluene	50 - 158	0 - 40	95	5.0	0.330	4.2	0.05
3-Nitroanaline	10 - 150	0 - 40	95	5.0	0.330	4.3	0.06
Acenaphthene	47 - 145	0 - 40	95	5.0	0.330	4.1	0.05
2,4-Dinitrophenol	D - 191	0 - 40	95	25.0	1.65	4.4	0.02
4-Nitrophenol	D - 132	0 - 40	95	5.0	0.330	2.7	0.11
Dibenzofuran	10 - 150	0 - 40	95	5.0	0.330	3.6	0.05
2,4-Dinitrotoluene	39 - 139	0 - 40	95	5.0	0.330	2.8	0.06
Diethyl phthalate	D - 114	0 - 40	95	5.0	0.330	3.9	0.04
Fluorene	59 - 121	0 - 40	95	5.0	0.330	3.1	0.04
4-Chlorophenylphenyl ether	25 - 158	0 - 40	95	5.0	0.330	3.1	0.04
4-Nitroanaline	10 - 150	0 - 40	95	5.0	0.330	4.2	0.05
2-Methyl-4,6-dinitrophenol	D - 181	0 - 40	95	25.0	1.65	3.5	0.02
N-Nitrosodiphenylamine	10 - 150	0 - 40	95	5.0	0.330	1.4	0.05
Azobenzene	10 - 150	0 - 40	95	5.0	0.330	33.5	0.05
4-Bromphenylphenyl ether	53 - 127	0 - 40	95	5.0	0.330	2.2	0.06
Hexachlorobenzene	D - 152	0 - 40	95	5.0	0.330	1.8	0.06
Pentachlorophenol	14 - 176	0 - 40	95	5.0	0.330	1.8	0.07
Phenanthrene	54 - 120	0 - 40	95	5.0	0.330	2.3	0.07
Anthracene	27 - 133	0 - 40	95	5.0	0.330	2.5	0.05
Carbazole	10 - 150	0 - 40	95	5.0	0.330	1.5	0.04
Di-n-butyl phthalate	1 - 118	0 - 40	95	5.0	0.330	1.9	0.05

nd- not determined

TABLE 15. (cont.)
EPA METHOD 8270
QC Acceptance Criteria and Detection Limit Summary

COMPOUNDS	Accuracy %R	Precision RPD	Completeness %	Water MRL (ug/L)	Soil MRL (mg/Kg)	Water MDL (ug/L)	Soil MDL (ug/Kg)
Fluoranthene	26 - 137	0 - 40	95	5.0	0.330	1.2	0.04
Pyrene	52 - 115	0 - 40	95	5.0	0.330	0.7	0.05
Butyl benzyl phthalate	D - 152	0 - 40	95	5.0	0.330	3.4	0.04
Benzo(a)anthracene	33 - 143	0 - 40	95	5.0	0.330	4.3	0.09
3,3'-Dichlorobenzidine	D - 262	0 - 40	95	25.0	1.65	2.7	0.10
Chrysene	17 - 168	0 - 40	95	5.0	0.330	1.2	0.04
bis(2-Ethylhexyl) phthalate	8 - 158	0 - 40	95	5.0	0.330	2.5	0.03
Di-n-octyl phthalate	4 - 146	0 - 40	95	5.0	0.330	3.7	0.04
Benzo(b)fluoranthene	24 - 159	0 - 40	95	5.0	0.330	1.6	0.07
Benzo(k)fluoranthene	11 - 162	0 - 40	95	5.0	0.330	2.7	0.05
Benzo(a)pyrene	17 - 163	0 - 40	95	5.0	0.330	1.1	0.04
Indeno(1,2,3-cd)pyrene	D - 171	0 - 40	95	5.0	0.330	1.4	0.03
Dibenzo(a,h)anthracene	D - 227	0 - 40	95	5.0	0.330	0.8	0.03
Benzo(g,h,i)perylene	D - 219	0 - 40	95	5.0	0.330	0.6	0.04
Arochlor 1016	10 - 150	0 - 50	95	10.0	0.5	nd	nd
Arochlor 1221	10 - 150	0 - 50	95	10.0	0.5	nd	nd
Arochlor 1232	10 - 150	0 - 50	95	10.0	0.5	nd	nd
Arochlor 1242	10 - 150	0 - 50	95	10.0	0.5	nd	nd
Arochlor 1248	10 - 150	0 - 50	95	10.0	0.5	nd	nd
Arochlor 1254	10 - 150	0 - 50	95	10.0	0.5	nd	nd
Arochlor 1260	D - 164	0 - 50	95	10.0	0.5	nd	nd

nd- not determined

TABLE 16.
EPA METHOD 524.2
QC Acceptance Criteria and Detection Limit Summary

Compounds	Accuracy % R	Precision RPD	Completeness	MRL (ug/L)	MDL (ug/L)
Dichlorodifluoromethane	70 - 130	0 - 20	95	0.50	0.18
Chloromethane	70 - 130	0 - 20	95	0.50	0.20
Vinyl chloride	70 - 130	0 - 20	95	0.50	0.16
Bromomethane	70 - 130	0 - 20	95	0.50	0.21
Chloroethane	70 - 130	0 - 20	95	0.50	0.25
Trichlorofluoromethane	70 - 130	0 - 20	95	0.50	0.15
1,1-Dichloroethene	70 - 130	0 - 20	95	0.50	0.12
Methylene chloride	70 - 130	0 - 20	95	0.50*	0.29*
trans-1,2-dichloroethene	70 - 130	0 - 20	95	0.50	0.14
1,1-Dichloroethane	70 - 130	0 - 20	95	0.50	0.14
cis-1,2-Dichloroethene	70 - 130	0 - 20	95	0.50	0.15
2,2-Dichloropropane	70 - 130	0 - 20	95	0.50	0.27
Bromochloromethane	70 - 130	0 - 20	95	0.50	0.25
Chloroform	70 - 130	0 - 20	95	0.50	0.20
1,1,1-Trichloroethane	70 - 130	0 - 20	95	0.50	0.12
Carbon tetrachloride	70 - 130	0 - 20	95	0.50	0.23
1,1-Dichloropropene	70 - 130	0 - 20	95	0.50	0.19
Benzene	70 - 130	0 - 20	95	0.50	0.13
1,2-Dichloroethane	70 - 130	0 - 20	95	0.50	0.18
Trichloroethene	70 - 130	0 - 20	95	0.50	0.14
1,2-Dichloropropane	70 - 130	0 - 20	95	0.50	0.22
Dibromomethane	70 - 130	0 - 20	95	0.50	0.19
Bromodichloromethane	70 - 130	0 - 20	95	0.50	0.12
cis-1,3-Dichloropropene	70 - 130	0 - 20	95	0.50	0.15
Toluene	70 - 130	0 - 20	95	0.50	0.16
trans-1,3-Dichloropropene	70 - 130	0 - 20	95	0.50	0.25
1,1,2-Trichloroethane	70 - 130	0 - 20	95	0.50	0.24
Tetrachloroethene	70 - 130	0 - 20	95	0.50	0.23
1,3-Dichloropropane	70 - 130	0 - 20	95	0.50	0.22
Dibromochloromethane	70 - 130	0 - 20	95	0.50	0.17
1,2-Dibromoethane	70 - 130	0 - 20	95	0.50	0.25
Chlorobenzene	70 - 130	0 - 20	95	0.50	0.15
1,1,1,2-Tetrachloroethane	70 - 130	0 - 20	95	0.50	0.19
Ethylbenzene	70 - 130	0 - 20	95	0.50	0.15
m&p-Xylene	70 - 130	0 - 20	95	0.50	0.11
o-Xylene	70 - 130	0 - 20	95	0.50	0.19
Styrene	70 - 130	0 - 20	95	0.50	0.25
Bromoform	70 - 130	0 - 20	95	0.50	0.19
Isopropylbenzene	70 - 130	0 - 20	95	0.50	0.13
1,1,2,2-Tetrachloroethane	70 - 130	0 - 20	95	0.50	0.18
Bromobenzene	70 - 130	0 - 20	95	0.50	0.13
1,2,3-Trichloropropane	70 - 130	0 - 20	95	0.50	0.09
n-Propylbenzene	70 - 130	0 - 20	95	0.50	0.15
2-Chlorotoluene	70 - 130	0 - 20	95	0.50	0.23
1,3,5-Trimethylbenzene	70 - 130	0 - 20	95	0.50	0.21
4-Chlorotoluene	70 - 130	0 - 20	95	0.50	0.19
tert-Butylbenzene	70 - 130	0 - 20	95	0.50	0.12
1,2,4-Trimethylbenzene	70 - 130	0 - 20	95	0.50	0.23
sec-Butylbenzene	70 - 130	0 - 20	95	0.50	0.14
1,3-Dichlorobenzene	70 - 130	0 - 20	95	0.50	0.25
p-Isopropyltoluene	70 - 130	0 - 20	95	0.50	0.21
1,4-Dichlorobenzene	70 - 130	0 - 20	95	0.50	0.30
n-Butylbenzene	70 - 130	0 - 20	95	0.50	0.25
1,2-Dichlorobenzene	70 - 130	0 - 20	95	0.50	0.30
1,2-Dibromo-3-chloropropane	70 - 130	0 - 20	95	0.50	0.42
1,2,4-Trichlorobenzene	70 - 130	0 - 20	95	0.50	0.35
Hexachlorobutadiene	70 - 130	0 - 20	95	0.50	0.28
Naphthalene	70 - 130	0 - 20	95	0.50	0.29
1,2,3-Trichlorobenzene	70 - 130	0 - 20	95	0.50	0.31

* Methylene chloride is a common laboratory contaminant. this detection limit value may not be routinely achieved under normal laboratory operations.

TABLE 17.
AROMATIC VOLATILE ORGANICS BY GC
(low level method)

COMPOUNDS	Accuracy %R	Precision RPD	Completeness	Water MRL (ug/L)	Soil MRL (ug/Kg)	Water MDL (ug/L)	Soil MDL (ug/Kg)
Benzene	39 - 150	0 - 20	95	0.4	2.0	0.078	0.37
Toluene	46 - 148	0 - 20	95	0.4	2.0	0.292	0.37
Chlorobenzene	55 - 135	0 - 20	95	0.4	2.0	0.095	0.48
Ethylbenzene	32 - 160	0 - 20	95	0.4	2.0	0.074	0.31
m&p-Xylene	50 - 150	0 - 20	95	0.4	2.0	0.135	0.78
o-Xylene	50 - 150	0 - 20	95	0.4	2.0	0.086	0.35
1,3-Dichlorobenzene	50 - 141	0 - 20	95	0.4	2.0	0.107	0.54
1 4-Dichlorobenzene	42 - 143	0 - 20	95	0.4	2.0	0.075	0.38
1,2-Dichlorobenzene	37 - 154	0 - 20	95	0.4	2.0	0.065	0.32

TOTAL PETROLEUM HYDROCARBONS

COMPOUNDS	Water Accuracy %R	Precision RPD	Soil Accuracy %R	Precision RPD	Completeness	Water MRL (mg/L)	Soil MRL (mg/Kg)	Water MDL (mg/L)	Soil MDL (mg/Kg)
TPH IR	81 - 112	0 - 6	82 - 108	0 - 8	95	1.0	4.0	0.07	3.6
TPH GC GRO	71 - 115	0 - 17	50 - 150	0 - 25	95	1.0	4.0	0.015	0.04
TPH GC DRO	48 -121	0 - 15	84 - 122	0 - 18	95	1.0	4.0	0.8	0.92
Total TPH GC	48 - 121	0 - 15	84 - 122	0 - 18	95	1.0	4.0	0.8	0.92

nd - Not determined.

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

COMPOUNDS	Accuracy %R	Precision RPD	Completeness	Water MRL (ug/L)	Water MDL (ug/L)	Soil MRL (ug/Kg)	Soil MDL (ug/Kg)
Aldrin	42 - 122	0 - 30	95	0.05	0.0044	0.05	0.95
alpha-BHC	37 - 134	0 - 30	95	0.05	0.0020	0.05	0.86
beta-BHC	17 - 147	0 - 30	95	0.05	0.0027	0.05	1.51
delta-BHC	19 - 140	0 - 30	95	0.05	0.0025	0.05	0.80
gamma-BHC (Lindane)	32 - 127	0 - 30	95	0.05	0.0021	0.05	0.80
4,4'-DDD	31 - 141	0 - 30	95	0.10	0.0081	3.3	2.10
4,4'-DDE	30 - 145	0 - 30	95	0.10	0.0071	3.3	2.42
4,4'-DDT	25 - 160	0 - 30	95	0.10	0.0074	3.3	2.25
Dieldrin	36 - 146	0 - 30	95	0.10	0.0073	3.3	2.09
Endosulfan I	45 - 153	0 - 30	95	0.05	0.0031	1.7	0.81
Endosulfan II	D - 202	0 - 30	95	0.10	0.0083	3.3	2.29
Endosulfan sulfate	26 - 144	0 - 30	95	0.10	0.0080	3.3	2.11
Endrin	30 - 147	0 - 30	95	0.10	0.0077	3.3	2.52
Endrin Aldehyde	25-150*	0 - 30	95	0.10	0.0089	3.3	2.13
Heptachlor	34 - 111	0 - 30	95	0.05	0.0021	1.7	0.99
Heptachlor epoxide	37 - 142	0 - 30	95	0.05	0.0026	1.7	0.39
Methoxychlor	25-150*	0 - 30	95	0.50	0.0193	17	11.5
Toxaphene	41 - 126	0 - 30	95	2.50	0.94	85	5.53
Chlordane	45 - 119	0 - 30	95	2.50	1.77	85	48.8
Aroclor 1016	50 - 114	0 - 30	95	1.5	0.612	33	9.05
Aroclor 1221	15 - 178	0 - 30	95	1.5	0.684	33	9.28
Aroclor 1232	10 - 215	0 - 30	95	1.5	0.632	33	15.1
Aroclor 1242	39 - 150	0 - 30	95	1.5	0.171	33	11.3
Aroclor 1248	38 - 158	0 - 30	95	1.5	0.850	33	9.32
Aroclor 1254	29 - 131	0 - 30	95	1.5	0.498	33	30.2
Aroclor 1260	8 - 127	0 - 30	95	1.5	0.430	33	2.23

TABLE 18.
METHODS 610 AND 8100
QC Acceptance Limits, Reporting Limits and Detection Limits

COMPOUNDS	Accuracy* %R	Precision* RPD	Completeness	Water MRL (ug/L)	Soil MRL (mg/Kg)	Water MDL** (ug/L)	Soil MDL** (ug/L)
Naphthalene	D - 122	0 - 50	95	1.00	0.33	0.42	0.30
Acenaphthylene	D - 139	0 - 50	95	1.00	0.33	0.65	0.27
Acenaphthene	D - 124	0 - 50	95	1.00	0.33	0.61	0.31
Fluorene	D - 142	0 - 50	95	1.00	0.33	0.71	0.31
Phenanthrene	D - 155	0 - 50	95	1.00	0.33	0.70	0.29
Anthracene	D - 126	0 - 50	95	1.00	0.33	0.76	0.31
Fluoranthene	14 - 123	0 - 50	95	1.00	0.33	0.67	0.24
Pyrene	D - 140	0 - 50	95	1.00	0.33	0.65	0.21
Benzo(a)anthracene	12 - 135	0 - 50	95	1.00	0.33	0.57	0.23
Chrysene	D - 199	0 - 50	95	1.00	0.33	0.59	0.26
Benzo(b)fluoranthene	6 - 150	0 - 50	95	1.00	0.33	0.48	0.27
Benzo(k)fluoranthene	D - 159	0 - 50	95	1.00	0.33	0.62	0.22
Benzo(a)pyrene	D - 128	0 - 50	95	1.00	0.33	0.52	0.23
Indeno(1,2,3-cd)pyrene	D - 116	0 - 50	95	1.00	0.33	0.79	0.18
Dibenzo(a,h)anthracene	D - 110	0 - 50	95	1.00	0.33	0.64	0.16
Benzo(ghi)perylene	D - 116	0 - 50	95	1.00	0.33	0.68	0.19

D = Detected; result must be greater than zero.

* - Accuracy and precision are the published acceptance criteria from EPA method 610 and 40 CFR part 136.

** - Detection limits listed were determined from laboratory data using a flame ionization detector.

METHOD 8121
QC Acceptance Limits, Reporting Limits and Detection Limits

	Completeness	% Recovery Acceptance Range for Water	RPD limits for Water	MRL Water µg/L	MDL Water µg/mL	% Recovery Acceptance Range for Soil	RPD limits for Soil	MRL Soil µg/Kg	MDL Soil µg/Kg
Benzal chloride	95	5 - 150	0 - 30	0.20	0.09	5 - 150	0 - 50	20.0	6.71
Benzotrichloride	95	5 - 150	0 - 30	0.20	0.07	5 - 150	0 - 50	20.0	4.30
Benzyl chloride	95	5 - 150	0 - 30	0.50	0.33	5 - 150	0 - 50	50.0	18.7
2-Chloronaphthalene	95	9 - 148	0 - 30	1.00	0.57	9 - 148	0 - 50	50.0	28.8
1,2-Dichlorobenzene	95	9 - 160	0 - 30	0.50	0.13	9 - 160	0 - 50	10.0	1.36
1,3-Dichlorobenzene	95	D - 150	0 - 30	0.50	0.13	D - 150	0 - 50	10.0	1.36
1,4-Dichlorobenzene	95	13 - 137	0 - 30	0.50	0.13	13 - 137	0 - 50	10.0	1.36
Hexachlorobenzene	95	15 - 159	0 - 30	0.20	0.06	15 - 159	0 - 50	10.0	1.84
Hexachlorobutadiene	95	D - 139	0 - 30	0.50	0.18	D - 139	0 - 50	10.0	2.50
alpha-BHC	95	5 - 150	0 - 30	0.20	0.07	5 - 150	0 - 50	10.0	1.84
beta-BHC	95	5 - 150	0 - 30	1.00	0.42	5 - 150	0 - 50	50.0	13.9
gamma-BHC	95	5 - 150	0 - 30	0.20	0.07	5 - 150	0 - 50	10.0	1.84
delta-BHC	95	5 - 150	0 - 30	1.00	0.42	5 - 150	0 - 50	50.0	13.9
Hexachlorocyclopentadiene	95	D - 111	0 - 30	0.50	0.18	D - 111	0 - 50	10.0	2.37
Hexachloroethane	95	8 - 139	0 - 30	0.50	0.13	8 - 139	0 - 50	10.0	1.36
Pentachlorobenzene	95	5 - 150	0 - 30	0.20	0.06	5 - 150	0 - 50	10.0	1.50
1,2,3,4-Tetrachlorobenzene	95	5 - 150	0 - 30	0.50	0.13	5 - 150	0 - 50	10.0	3.82
1,2,3,5-Tetrachlorobenzene	95	5 - 150	0 - 30	0.50	0.17	5 - 150	0 - 50	20.0	6.00
1,2,4,5-Tetrachlorobenzene	95	5 - 150	0 - 30	0.50	0.17	5 - 150	0 - 50	20.0	6.00
1,2,4-Trichlorobenzene	95	5 - 149	0 - 30	0.20	0.09	5 - 149	0 - 50	20.0	6.71
1,2,3-Trichlorobenzene	95	5 - 149	0 - 30	0.50	0.13	5 - 149	0 - 50	10.0	4.11
1,3,5-Trichlorobenzene	95	5 - 149	0 - 30	0.50	0.28	5 - 149	0 - 50	50.0	12.6

TABLE 19.
METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP-AES)
WATER EPA METHOD 200.7 AND SW846 METHOD 6010
SOIL/SEDIMENT SW846 METHOD 6010

Element	Water Accuracy %R	Precision RPD	Soil Accuracy %R	Precision RPD	Completeness %	Water MRL (mg/L)	Soil MRL (mg/Kg)	Water MDL (mg/L)	Soil MDL (mg/Kg)
Aluminum	75 - 125	0 - 20	75 - 125	0 - 20	95	0.200	10.00	0.014	1.62
Antimony	75 - 125	0 - 20	75 - 125	0 - 20	95	0.060	5.00	0.043	3.84
Arsenic	69 - 116	0 - 20	58 - 127	0 - 20	95	50	5.00	0.018	4.71
Barium	67 - 118	0 - 20	62 - 129	0 - 20	95	0.005	0.50	0.0006	0.11
Beryllium	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	0.50	0.011	0.11
Cadmium	67 - 115	0 - 20	67 - 131	0 - 20	95	0.005	0.50	0.0017	0.18
Calcium	75 - 125	0 - 20	75 - 125	0 - 20	95	2.00	250	0.028	6.94
Chromium	67 - 112	0 - 20	61 - 115	0 - 20	95	0.005	0.50	0.0015	0.09
Cobalt	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	0.50	0.0008	0.16
Copper	75 - 125	0 - 20	75 - 125	0 - 20	95	0.010	1.00	0.0012	0.75
Iron	75 - 125	0 - 20	75 - 125	0 - 20	95	0.100	5.0	0.004	2.39
Lead	68 - 111	0 - 20	60 - 118	0 - 20	95	0.005	1.00	0.004	0.64
Lithium	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	0.50	0.0002	0.11
Magnesium	75 - 125	0 - 20	75 - 125	0 - 20	95	5.00	250	0.018	5.59
Manganese	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	0.25	0.7	0.32
Molybdenum	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	1.00	0.002	0.47
Nickel	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	0.25	0.004	0.37
Potassium	75 - 125	0 - 20	75 - 125	0 - 20	95	5.00	5.0	0.027	4.65
Selenium	70 - 123	0 - 20	67 - 140	0 - 20	95	0.050	2.50	0.018	3.91
Silver	61 - 123	0 - 20	68 - 130	0 - 20	95	0.005	0.25	0.0009	0.16
Sodium	75 - 125	0 - 20	75 - 125	0 - 20	95	5.00	1.0	0.044	8.83
Thallium	75 - 125	0 - 20	75 - 125	0 - 20	95	0.050	2.50	0.019	2.32
Vanadium	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	0.25	0.001	0.44
Zinc	75 - 125	0 - 20	75 - 125	0 - 20	95	0.005	1.0	0.0009	1.75

note- The estimated detection limits as shown are taken from Inductively Coupled Plasma Atomic Emission Spectroscopy-Prominent Lines, EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

MERCURY
WATER EPA METHOD 245.1 AND SW846 METHOD 7470
SOIL/SEDIMENT EPA METHOD 245.5 AND SW846 METHOD 7471

Element	Water Accuracy %R	Water Precision RPD	Soil Accuracy %R	Soil Precision RPD	Completeness %	Water MRL (ug/L)	Soil MRL (mg/Kg)	Water MDL (ug/L)	Soil MDL (mg/Kg)
Mercury	71 - 109	0 - 20	75 - 112	0 - 20	95	0.2	0.2	0.07	0.01

TABLE 20.
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR WATER SAMPLES

CHEMICAL ANALYSES	MINIMUM VOLUME (mL)*	PLASTIC OR GLASS	PRESERVATION	HOLDING TIME
Acidity	100	P, G	cool, 4°C	14 days
Alkalinity	100	P,G	cool, 4°C	14 days
Ammonia	100	P,G	cool, 4°C sulfuric to pH<2	28 days
BOD 5 day	1000	P,G	cool, 4°C	48 hours
Total Organic Carbon (TOC)	50	P,G	cool, 4°C sulfuric to pH<2	28 days
Chemical Oxygen Demand	50	P,G	cool, 4°C sulfuric to pH<2	28 days
Chloride	100	P,G	none	28 days
Chromium, hexavalent	100	P,G	cool, 4°C	24 hours
Color	50	P,G	cool, 4°C	48 hours
Conductance	100	P,G	cool, 4°C	28 days
Cyanide		P,G	cool, 4°C	14 days
free	100		NaOH to pH>12	
total	*500*			
total & ammenable	1000			
Fecal Coliform	100	P,G	cool, 4°C	6 hours
Fluoride	100	P	none	28 days
Hardness	100	P,G	none	6 months
Metals (except mercury) (200.7, 200.8, 6010 & 6020)	500	P,G	nitric to pH<2	6 months
Mercury (245.1, 7470)	500	P,G	nitric to pH<2	28 days
Nitrogen		P,G	cool, 4°C	28 days
total Kjeldahl	100		sulfuric to pH<2	
Nitrate	100			
Nitrite	100			
Oil and Grease (413.1)	1000	G only	cool, 4°C *sulfuric or HCl to pH<2*	28 days
Organic Analytes				
Volatiles	40	G only	cool, 4°C HCl to pH<2	14 days
BTEX, VOC's, TPH/GRO (8020, 8260A, 8260B 8015mod)	(3 per sample)	PTFE septa for VOA's		
Volatiles (524.2)	40 (3 per sample)	G only PTFE septa for VOA's	cool, 4°C,3 mg of Na2S203	14 days
EDB, DBCP (8011)	40 (3 per sample)	G only PTFE septa for VOA's	cool, 4°C, 4 drops of 10% Na2S203	14 days
Semi-volatiles (505)	40 (3 per sample)	G only PTFE septa for VOA's	cool, 4°C, 3 mg of Na2S203.	7 days to extr.
TPH/DRO	1000	G only	cool, 4°C	7 days to extr.
Semi-volatiles (8270B)	1000	with PTFE	cool, 4°C	40 days after
Semi-volatiles (525.2)	1000	lined cap	cool, 4°C, 50 mg of Na2SO3 HCl to pH<2	14 days
Pesticides/PCB's (8081)	1000		cool, 4°C	7 days to extr.
Herbicides (8150, 8151)	1000		cool, 4°C	40 days after
PH (150.1)	100	P,G	none	immediately
Phenolics	100	G only	cool, 4°C sulfuric to pH<2	28 days
Phosphorous				
ortho phosphate	50	P,G	filter on site	48 hours
total phosphate	50	P,G	cool, 4°C	48 hours

TABLE 20. (cont.)
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR WATER SAMPLES

CHEMICAL ANALYSES	MINIMUM VOLUME (mL)*	PLASTIC OR GLASS	PRESERVATION	HOLDING TIME
Solids		P,G	cool, 4°C	
dissolved	100			48 hours
suspended	250			7 days
total	250			7days
volatile	250			7 days
settleable	1000			48 hours
Sulfate	50	P,G	cool, 4°C	28 days
Sulfide	500	P,G	cool, 4°C 2 mL ZnOAc and NaOH to pH>9	7 days
Sulfite	50	P,G	none	immediately
Total Recoverable Petroleum Hydrocarbons (418.1)	1000	G only with Teflon lined cap	cool, 4°C HCl to pH <2	7 days
Turbidity	100	P,G	cool, 4°C	48 hours

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR SOIL SAMPLES

CHEMICAL ANALYSES	MINIMUM CONTAINER SIZE (mL)*	PLASTIC OR GLASS	PRESERVATION	HOLDING TIME
Nutrients/TOC	400	P	cool to 4° C	14 days
Metals (except mercury) (6010 & 6020)	250	P or G	none required	6 months
Mercury	250	P or G	cool to 4° C	28 days
Volatile Organics (SW846 8240, 8260, 8020, 8010)	125	G only with PTFE lined cap	dark, cool to 4° C	14 days
Volatile Organics Low Level (SW846 8260B/5035)	3 x 40 mL	G only with PTFE lined cap	5ml of 20% sodium bisulfate, dark, cool to 4° C	14 days
Volatile Organics High Level (SW846 8260B/5035)	1 X 40 mL	G only with PTFE lined cap	10 mL of purge and trap grade methanol, dark, cool to 4° C	14 days
Volatile Organics percent moisture (SW846 8260B/5035)	1 X 40 mL	G only with PTFE lined cap	dark, cool to 4° C	14 days
Semi-volatile organics, TPH, pesticides, PCB's, etc. (SW846 8270, 8080)	250	G only with PTFE lined cap	dark, cool to 4° C	14 days before extraction, 40 days after extraction
BTEX (SW846 8020)	125	G only with PTFE lined cap	dark, cool to 4° C	14 days
TPH by GC (SW846 8015 modified)	125	G only with PTFE lined cap	dark, cool to 4° C	14 days
TPH by IR (EPA 418.1)	125	G only with PTFE lined cap	dark, cool to 4° C	7 days

** - These are recommended minimum volumes for individual parameters. Some parameters with similar requirements may be combined in a larger container. Please call the laboratory for advice if you wish to combine sampling containers for multiple analyses.*

TABLE 21.
GEO Analytical Equipment List

Equipment	Manufacturer	Model	Number Owned
Gas Chromatograph	Hewlett Packard	5890 Series II	4
Gas Chromatograph	Varian	3400 CX	2
Gas Chromatograph/ Mass Spectrometer	Varian	Saturn GC/MS/MS	4
~~Automatic Sampler~~	~~Dynatech~~	~~PTA WS30~~	1
Vial Autosampler	Varian	8100	1
Vial Autosampler	Varian	8200	1
ICP	Varian	Liberty 100	1
ICP/MS	Varian	Ultramass	1
Microwave Digestor	CEM	MDS 2100	1
Vial Autosampler	Hewlett Packard	7673	2
Autosampler	Varian	SPS-5	1
Data System-Chemstation	Hewlett Packard	3365	4
Photoionization detector	O.I. Analytical	4430	2
Flame Ionization detector	O.I. Analytical	4410	3
Flame Ionization detector	Hewlett Packard		2
Purge and Trap	Tekmar	LSC 2000	3
Purge and Trap	Tekmar	LSC 3000	1
Automatic Sampler	Dynatech	Model 5100 Archon	4
Sample Concentrator	Zymark	Turbovap 200	2
Infrared Spectrophotometer	Perkin Elmer	1605	1
Ultrasonic Disruptor	Tekmar	TM375	1
Ultrasonic Disruptor	Virtis	Virsonic 300	1
Pulse Module	Virtis		1
Top Loader Balance	Sartorius	PT120	3
Analytical Balance	Sartorius	A200S	1
Analytical Balance	Sartorius	A210S	1
Analytical Mill	Tekmar	A-10	1
Ultrasonic Bath	Branson	2200	1

TABLE 21.
GEO Analytical Equipment List (Continued)

Equipment	Manufacturer	Model	Number Owned
Computer	PC Systems	Pentium 233	1
Computer	PC Systems	Pentium 200	6
Computer	PC Systems	Pentium 150	1
Computer	PC Systems	Pentium 133	4
Computer	PC Systems	Pentium 200	1
Computer	PC Systems	Pentium 466	1
Computer	PC Systems	Pentium 366	2
Computer	PC Systems	Pentium 333	2
Computer	Digital Equipment Corp	Pentium 166	1
Computer	Compaq	Pentium 400	1
Laser Printer	Panasonic	KX-P4420	2
Laser Printer	Panasonic	KX-P4430	1
Laser Printer	Panasonic	KX-P4440	2
Laser Printer	Panasonic	KX-P4400	1
Laser Printer	Minolta	PageWorks 20	1
Laboratory Fume Hood	Labconco	Protector 72	3
Wrist Shaker	Lab Line	MultiWrist	1
Drying Oven	Scientific Products		3
pH Meter	Orion	Model 420A	1
Ultrasonic Nebulizer	Varian	Model U-5000 AT	1
Hot Plate	Lindberg	Blue M 53025	1
Vapor Generation Accessory	Varian	Model 76	1
Mercury Analyzer System	Cetac	M6000-A	1
Zero Headspace Extractor	Millipore		5
Haz Waste Filtration System	Millipore		1
Environmental Rotator	Environ. Express	LE6000	1
Water Bath	VWR Scientific	1245	1

Table A-1
QUALITY CONTROL FOR METALS METHODS

	GAM 200.8	GAM 6020 Water	GAM 6020 Soil
Frequency of method blanks	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch	one per every 20 samples or analytical batch
Initial Calibration Blank Verification (ICB)	following instrument calibration and must be less than the minimum reporting limit or less than 10% of the sample concentration	following instrument calibration and must be less than the minimum reporting limit or less than 10% of the sample concentration	following instrument calibration and must be less than the minimum reporting limit or less than 10% of the sample concentration
Initial Calibration Verification (ICV)	following instrument calibration verify calibration with an independently prepared check standard. %R = 95 – 105%	following instrument calibration verify calibration with an independently prepared check standard. %R = 95 – 105%	following instrument calibration verify calibration with an independently prepared check standard. %R = 95 – 105%
Laboratory Control Samples	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.
Frequency of MS and MSD's	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.	one MS/MSD per 20 samples or analytical batch.
Accuracy and Precision	compound dependent see Table A-4.	compound dependent see Table A-4.	compound dependent see Table A-4.
Internal Standards	yes	yes	yes
Initial Calibration	3 levels and a blank $r^2 \geq 0.995$	3 levels and a blank $r^2 \geq 0.995$	3 levels and a blank $r^2 \geq 0.995$
Continuing Calibration Blank Verification (CCB)	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration	after every ten samples and at the end of each sequence and must be less than the minimum reporting limit or less than 10% of the sample concentration
Continuing Calibration Verification (CCV)	after every ten samples and at the end of each sequence and must be within 90-100% of initial calibration	after every ten samples and at the end of each sequence and must be within 90-100% of initial calibration	after every ten samples and at the end of each sequence and must be within 90-100% of initial calibration
Interference Check Samples (ICS)	n/a	Run at the beginning of each sequence. ICSA target analytes must be below reporting limit or less than 10% of the sample concentration. ICSAB target analytes must be $\pm$ 20% of expected.	n/a.
Mass Calibration and Performance Parameters	Table A-5.	Table A-5.	Table A-5.
Detection Limits	Table A-4	Table A-4	Table A-4

TABLE A-2.
QUALITY CONTROL FOR ORGANIC CONTAMINANTS

	GAM 505	GAM 525.2	GAM 8011	GPBM 8015	GPBM 8260	GAM 8260B
Frequency of method blanks	one per every 20 samples or 24 hours. Whichever is more frequent.	one per every 20 samples or 12 hours. Whichever is more frequent.	one per analytical batch or every 20 samples.	one per analytical batch or every 20 samples.	one per analytical batch or every 20 samples.	one per analytical batch or every 20 samples.
Frequency of Reagent blanks	One per 20 samples or 24 hours . Whichever is more frequent.	One per 20 samples or 12 hours . Whichever is more frequent.	One per ten analytical samples.	One per ten analytical samples.	One per 12 hour clock	One per 12 hour clock
Surrogates	no	% R = 70 - 130	no	no	1,2-dicloroethane-d4 placed in all samples, blanks, spikes, duplicates. Acceptable recovery range. Table A-3	placed in all samples, blanks, spikes, duplicates. %R ranges in Table A-3.
Laboratory Control Samples	one LFB per 20 samples or 24 hours.	one LFB per 20 samples or 12 hours.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.
Frequency of MS and MSD	one LFM & LFMD per 20 samples or 24 hours. Same conc as LFB.	one LFM & LFMD per 20 samples or 24 hours. Same conc as LFB	one MS/MSD per analytical batch or every 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.
Accuracy Precision	% R = 70 - 130 RPD < 30%	% R = 70 - 130 RPD < 30%	%R=50 -150 RPD < 50%	%R=50 - 150 RPD < 30%	compound dependent see Table 18.	compound dependent see Table 7A.
Internal standards	no	ISTD's in the CCV should be >70% CCV to CCV, but must be >50% of initial calib.	no	no	ISTD's must be < +100% and >50% of the last CCV.	ISTD's must be < +100% and >50% of the last CCV.
Initial calibration	3 point minimum	5 point minimum, 6 point recommended	5 point minimum ≤ 20% RSD for average RF. r^2 ≥ 0.990	5 point minimum ≤ 20% RSD for average RF. r^2 ≥ 0.990	5 point minimum ≤ 30% RSD for average RF. r^2 ≥ 0.990	5 point minimum ≤ 30% RSD for average RF of CCC's, < 15% RSD for all others or r^2 ≥ 0.990
Continuing Calibration Check	calibration standard run every 10 samples and must be within ± 20 % diff. of average RF	calibration standard run every 12 hours and must be within ± 30 % diff. of average RF or ±30% of value	calibration standard run every 10 samples and must be within ± 40 % diff. of average RF	calibration standard run every 10 samples and must be within ± 15 % diff. Of average RF	calibration standard run every 10 samples and must be within ± 15 % diff of average RF	calibration standard run every 12 hours and must be within ± 20 % diff. of average RF
Ending Calibration Check	no	no	run at the end of each sequence and must be within ± 40% diff of average RF	run at the end of each sequence and must be within ± 15 % diff. of average RF	run at the end of each sequence and must be within ± 15 % diff. of average RF	n/a
Other	Daily PEM mix < 20% Endrin breakdown. QCS sample run at least quarterly.	Daily PEM mix < 20% Endrin breakdown. QCS sample run at least quarterly.	none	none	none	none
Tuning parameters	no	DFTPP criteria in table A-5. every 12 hours while samples are being run.	no	no	no	BFB to criteria in Table A-5, every 12 hours while samples are being run.
Detection limits	Table A-3	Table A-3	Table A-3	Table A-3.	Table A-3.	Table A-3.

Table A-2
QUALITY CONTROL FOR ORGANIC CONTAMINANTS

	GAM 505	GAM 525.2	GAM 8011	GPBM 8015	GPBM 8260	GAM 8260B
Frequency of method blanks	one per every 20 samples or 24 hours. Whichever is more frequent.	one per every 20 samples or 12 hours. Whichever is more frequent.	one per analytical batch or every 20 samples.	one per analytical batch or every 20 samples.	one per analytical batch or every 20 samples.	one per analytical batch or every 20 samples.
Frequency of Reagent blanks	One per 20 samples or 24 hours . Whichever is more frequent	One per 20 samples or 12 hours . Whichever is more frequent.	One per ten analytical samples.	One per ten analytical samples.	One per 12 hour clock	One per 12 hour clock
Surrogates	no	% R = 70 - 130	no	no	1,2-dicloroethane-d4 placed in all samples, blanks, spikes, duplicates. Acceptable recovery range. Table A-3	placed in all samples, blanks, spikes, duplicates. %R ranges in Table A-3.
Laboratory Control Samples	one LFB per 20 samples or 24 hours.	one LFB per 20 samples or 12 hours.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.	one LCS per 20 samples or analytical batch.
Frequency of MS and MSD	one LFM & LFMD per 20 samples or 24 hours. Same conc as LFB.	one LFM & LFMD per 20 samples or 24 hours. Same conc as LFB	one MS/MSD per analytical batch or every 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples.	one MS/MSD per every analytical batch or 20 samples
Accuracy Precision	% R = 70 - 130 RPD < 30%	% R = 70 - 130 RPD < 30%	%R=50 -150 RPD < 50%	%R=50 - 150 RPD < 30%	compound dependent see Table 18.	compound dependent see Table 7A.
Internal standards	no	ISTD's in the CCV should be >70% CCV to CCV, but must be >50% of initial calib.	no	no	ISTD's must be < +100% and >50% of the last CCV.	ISTD's must be < +100% and >50% of the last CCV.
Initial calibration	3 point minimum	5 point minimum, 6 point recommended	5 point minimum ≤ 20% RSD for average RF. r^2 ≥ 0.990	5 point minimum ≤ 20% RSD for average RF r^2 ≥ 0.990	5 point minimum ≤ 30% RSD for average RF. r^2 ≥ 0.990	5 point minimum ≤ 30% RSD for average RF of CCC's, < 15% RSD for all others or r^2 ≥ 0.990
Continuing Calibration Check	calibration standard run every 10 samples and must be within ± 20 % diff. of average RF	calibration standard run every 12 hours and must be within ± 30 % diff. of average RF or ±30% of value	calibration standard run every 10 samples and must be within ± 40 % diff. of average RF	calibration standard run every 10 samples and must be within ± 15 % diff. Of average RF	calibration standard run every 10 samples and must be within ± 15 % diff. of average RF	calibration standard run every 12 hours and must be within ± 20 % diff. of average RF
Ending Calibration Check	no	no	run at the end of each sequence and must be within ± 40% diff of average RF	run at the end of each sequence and must be within ± 15 % diff of average RF	run at the end of each sequence and must be within ± 15 % diff. of average RF	n/a
Other	Daily PEM mix < 20% Endrin breakdown. QCS sample run at least quarterly	Daily PEM mix < 20% Endrin breakdown. QCS sample run at least quarterly	none	none	none	none
Tuning parameters	no	DFTPP criteria in table A-5, every 12 hours while samples are being run	no	no	no	BFB to criteria in Table A-5, every 12 hours while samples are being run.
Detection limits	Table A-3	Table A-3	Table A-3	Table A-3	Table A-3.	Table A-3.

Table A-3

GAM 525.2

METHOD DETECTION LIMITS (MDLS) FOR ANALYTES FROM REAGENT WATER

#	Compound	Mean		%R	Acceptance Limits	%RSD	Precision Limits	MDL (UG/L)	MRL (UG/L)
1	Naphthalene	4.589		92%	70 - 130	3%	0 - 30	0.09	0.5
2	Hexachlorocyclopentadiene	4.120		82%	70 - 130	11%	0 - 30	0.09	0.5
3	2,6-Dinitrotoluene	4.986		100%	70 - 130	2%	0 - 30	0.11	0.5
4	Acenaphthylene	3.986		80%	70 - 130	2%	0 - 30	0.11	0.5
5	Acenaphthene	4.038		81%	70 - 130	2%	0 - 30	0.11	0.5
6	2,4-Dinitrotoluene	5.146		103%	70 - 130	2%	0 - 30	0.11	0.5
7	Propachlor	4.702		94%	70 - 130	3%	0 - 30	0.09	0.5
8	Fluorene	4.290		86%	70 - 130	5%	0 - 30	0.11	0.5
9	Hexachlorobenzene	4.748		95%	70 - 130	4%	0 - 30	0.07	0.5
10	Simazine	5.194		104%	70 - 130	7%	0 - 30	0.13	0.5
11	Atrazine	4.894		98%	70 - 130	2%	0 - 30	0.12	0.5
12	Pentachlorophenol	5.784		116%	70 - 130	3%	0 - 30	0.10	0.5
13	gamma-BHC	5.010		100%	70 - 130	6%	0 - 30	0.12	0.5
14	Phenanthrene	4.854		97%	70 - 130	4%	0 - 30	0.06	0.5
15	Anthracene	4.485		90%	70 - 130	2%	0 - 30	0.11	0.5
16	Alachlor	5.090		102%	70 - 130	4%	0 - 30	0.10	0.5
17	Metribuzin	4.560		91%	70 - 130	6%	0 - 30	0.12	0.5
18	Heptachlor	5.013		100%	70 - 130	3%	0 - 30	0.08	0.5
19	Cyanazine	5.440		109%	70 - 130	5%	0 - 30	0.13	0.5
20	Metolachlor	4.982		100%	70 - 130	4%	0 - 30	0.15	0.5
21	Aldrin	4.850		97%	70 - 130	0%	0 - 30	0.08	0.5
22	Heptachlor epoxide	5.171		103%	70 - 130	3%	0 - 30	0.08	0.5
23	Fluoranthene	5.005		100%	70 - 130	4%	0 - 30	0.09	0.5
24	Butachlor	4.888		98%	70 - 130	6%	0 - 30	0.13	0.5
25	alpha-Chlordane	4.810		96%	70 - 130	5%	0 - 30	0.14	0.5
26	gamma-Chlordane	4.982		100%	70 - 130	5%	0 - 30	0.08	0.5
27	trans-Nonachlor	5.208		104%	70 - 130	2%	0 - 30	0.10	0.5
28	Dieldrin	4.701		94%	70 - 130	3%	0 - 30	0.08	0.5
29	Dieldrin	4.059		81%	70 - 130	4%	0 - 30	0.14	0.5
30	Endrin	4.632		93%	70 - 130	6%	0 - 30	0.17	0.5
31	bis(2-Ethylhexyl)adipate	4.504		90%	70 - 130	10%	0 - 30	0.12	0.5
32	Methoxychlor	3.713		74%	70 - 130	0%	0 - 30	0.03	0.5
33	bis(2-Ethylhexyl)phthalate	4.630		93%	70 - 130	7%	0 - 30	0.08	1.0
34	Benz(a)anthracene	4.521		90%	70 - 130	1%	0 - 30	0.04	0.5
35	Chrysene	4.266		85%	70 - 130	2%	0 - 30	0.03	0.5
36	Benzo(b)fluoranthene	4.674		93%	70 - 130	2%	0 - 30	0.10	0.5
37	Benzo(k)fluoranthene	4.235		85%	70 - 130	4%	0 - 30	0.07	0.5
38	Benzo(a)pyrene	4.470		89%	70 - 130	1%	0 - 30	0.10	0.5
39	Indeno(1,2,3-cd)pyrene	4.621		92%	70 - 130	3%	0 - 30	0.13	0.5
40	Dibenz(a,h)anthracene	4.544		91%	70 - 130	2%	0 - 30	0.17	0.5
41	Benzo(g,h,i)perylene	4.459		89%	70 - 130	5%	0 - 30	0.17	0.5

Table A-3 (cont.)
GAM 505
METHOD DETECTION LIMITS (MDLS) FOR ANALYTES FROM REAGENT WATER

COMPOUND	Accuracy Limits	RPD Limits	STD	MDL	MRL
Hexachlorocyclopentadiene	70 - 130	0 - 30	0.0060	0.0190	0.2
Hexachlorobenzene	70 - 130	0 - 30	0.0017	0.0052	0.2
Simazine	70 - 130	0 - 30	2.5069	7.8718	50
Atrazine	70 - 130	0 - 30	1.0800	3.3912	50
gamma-BHC (Lindane)	70 - 130	0 - 30	0.0033	0.0103	0.2
Heptachlor	70 - 130	0 - 30	0.0065	0.0204	0.2
Alachlor	70 - 130	0 - 30	0.0442	0.1389	2
Aldrin	70 - 130	0 - 30	0.0071	0.0224	0.2
Heptachlor epoxide	70 - 130	0 - 30	0.0110	0.0345	0.2
gamma-Chlordane	70 - 130	0 - 30	0.0024	0.0076	0.2
alpha-Chlordane	70 - 130	0 - 30	0.0024	0.0074	0.2
cis-Nonachlor	70 - 130	0 - 30	0.0028	0.0088	0.2
Dieldrin	70 - 130	0 - 30	0.0031	0.0097	0.2
Endrin	70 - 130	0 - 30	0.0044	0.0139	0.2
trans-Nonachlor	70 - 130	0 - 30	0.0046	0.0144	0.2
Methoxychlor	70 - 130	0 - 30	0.0439	0.1378	1

GAM 8011
QC Acceptance Criteria and Detection Limit Summary

Analyte	Accuracy %R	Precision RPD	Completeness %	MDL (µg/L)	MRL (µg/L)
EDB	50 - 150	0 - 50	95	0.039	0.05
DBCP	50 - 150	0 - 50	95	0.047	0.20

GAM 8260A
QC Acceptance Criteria and Detection Limit Summary

COMPOUNDS	Accuracy %R	Precision RPD	Completeness %	Water MRL (ug/L)	Soil MRL (ug/Kg)	Water MDL (ug/L)	Soil MDL (ug/Kg)
Propyl acetate	40 - 160	0 - 30	95	10	10	0.16	0.25
n-Hexane	40 - 160	0 - 30	95	5.0	5.0	0.46	0.43

GPBM 8015
Precision and Accuracy Limits for LCS , MS and MSD

COMPOUNDS	Completeness	Water MDL (mg/L)	Water MRL (mg/L)	Accuracy %R	Precision RPD	Soil MRL (mg/Kg)	Soil MDL (mg/Kg)
Propylene glycol	95	1.22	10.0	50 - 150	40	10.0	

Table A-3 (Cont.)

GAM 8260B
Acceptance Limits for Laboratory Control Samples, Matrix Spikes, and Spike Duplicates on Volatile Organics by GC/MS

COMPOUNDS	Water Accuracy %R	Water Precision RPD	Soil Accuracy %R	Soil Precision RPD	HL Soil Accuracy %R	HL Soil Precision RPD
1,1-Dichloroethene	67 - 149	14	36 - 156	19	59-172	22
Trichloroethene	67 - 131	12	50 - 119	21	66-142	21
Benzene	81 - 124	11	55 - 115	21	62-137	24
Toluene	54 - 138	12	54 - 117	19	59-139	21
Chlorobenzene	71 - 128	13	51 - 124	21	60-133	21

GAM 8260B SURROGATE RECOVERIES

METHOD 8240/8260A Surrogates	Acceptable Range for Water	Acceptable Range for Soil	Acceptable Range for High Level Soil
1,2-Dichloroethane d4	84 - 123	91 - 126	59 - 141
Toluene d8	91 - 117	66 - 136	56 - 152
Bromofluorobenzene	85 - 121	67 - 123	46 - 152

GPBM 8260A SURROGATE RECOVERIES

METHOD 8240/8260A Surrogates	Acceptable Range for Water	Acceptable Range for Soil	Acceptable Range for High Level Soil
1,2-Dichloroethane d4	84 - 123	91 - 126	59 - 141

TABLE A-4

METALS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (ICP-MS)
WATER EPA METHOD 200.8 AND SW846 METHOD 6020
SOIL/SEDIMENT SW846 METHOD 6020

Element	Water Accuracy %R	Precision RPD	Soil Accuracy %R	Precision RPD	Completeness %	Water MRL (ug/L)	Soil MRL (mg/Kg)	Water MDL (ug/L)	Soil MDL (mg/Kg)
Aluminum	75 - 125	0 - 20	75 - 125	0 - 20	95	200	20.0	1.583	0.1585
Antimony	75 - 125	0 - 20	75 - 125	0 - 20	95	6	1.5	0.093	0.0093
Arsenic	75 - 125	0 - 20	75 - 125	0 - 20	95	25	3.0	0.517	0.0517
Barium	75 - 125	0 - 20	75 - 125	0 - 20	95	15	3.0	0.471	0.0471
Beryllium	75 - 125	0 - 20	75 - 125	0 - 20	95	4	0.5	0.101	0.0101
Cadmium	75 - 125	0 - 20	75 - 125	0 - 20	95	5	1.0	0.120	0.0120
Calcium	75 - 125	0 - 20	75 - 125	0 - 20	95				
Chromium	75 - 125	0 - 20	75 - 125	0 - 20	95	5	1.0	0.230	0.0230
Cobalt	75 - 125	0 - 20	75 - 125	0 - 20	95	5	1.0	0.083	0.0083
Copper	75 - 125	0 - 20	75 - 125	0 - 20	95	5	1.0	0.487	0.0487
Iron	75 - 125	0 - 20	75 - 125	0 - 20	95				
Lead	75 - 125	0 - 20	75 - 125	0 - 20	95	4	0.5	0.158	0.0158
Lithium	75 - 125	0 - 20	75 - 125	0 - 20	95				
Magnesium	75 - 125	0 - 20	75 - 125	0 - 20	95				
Manganese	75 - 125	0 - 20	75 - 125	0 - 20	95	5	1.0	0.164	0.0164
Molybdenum	75 - 125	0 - 20	75 - 125	0 - 20	95				
Nickel	75 - 125	0 - 20	75 - 125	0 - 20	95	15	3.0	0.557	0.0557
Potassium	75 - 125	0 - 20	75 - 125	0 - 20	95				
Selenium	75 - 125	0 - 20	75 - 125	0 - 20	95	25	3.0	0.630	0.0630
Silver	75 - 125	0 - 20	75 - 125	0 - 20	95	5	0.5	0.074	0.0074
Sodium	75 - 125	0 - 20	75 - 125	0 - 20	95				
Thallium	75 - 125	0 - 20	75 - 125	0 - 20	95	2	0.5	0.061	0.0061
Vanadium	75 - 125	0 - 20	75 - 125	0 - 20	95	5	1.0	0.130	0.0130
Zinc	75 - 125	0 - 20	75 - 125	0 - 20	95	15	3.0	2.649	0.2649

note - The estimated detection limits as shown are taken from Inductively Coupled Plasma Atomic EmissionSpectroscopy-Prominent Lines, EPA-600/4-79-017. They are given as a guide for an instrumental limit.The actual method detection limits are sample dependent and may vary as the sample matrix varies.

TABLE A-5
MASS SPECTROMETER TUNING
ICP-MS METHODS

MASS CALIBRATION AND SYSTEM PERFORMANCE TEST CRITERIA

Mass Calibration*

	EPA 200.8	SW846 6020
Mass shift limit	< or = 1 amu	< or = 1 amu

Performance Test*

	EPA 200.8	SW846 6020
Replicates Required	minimum of 5	minimum of 4
Instrument Stability Requirement	RSD < or = 5%	RSD < 5%
Resolution Requirements	approximately 0.75 amu at 5% peak height recommended	0.9 amu full width at 10% peak height required
Ratio CeO/Ce	< 3% recommended	< 3% recommended
Ratio Ba++/Ba	< 5% recommended	< 5% recommended
Monitor Background at Mass	228 or 220	228 or 220

- Mass Calibration and Performance Test use tuning solution with analytes covering the entire mass range at a concentration of 100 ug/L.

GC/MS METHODS

ION ABUNDANCE TUNING CRITERIA FOR 4-BROMOFLUOROBENZENE (BFB)
Criteria for GAM 8260B and GPBM 8260

m/e	RELATIVE ABUNDANCE CRITERIA
50	15.0 - 40.0 % of mass 95.
75	30.0 - 60.0 % of mass 95.
95	Base peak, 100 % relative abundance.
96	5.0 - 9.0 % of mass 95.
173	Less than 2.0 % of mass 174.
174	Greater than 50.0 % of mass 95.
175	5.0 - 9.0 % of mass 174.
176	95.0 % but < 101.0 % of mass 174.
177	5.0 - 9.0 % of mass 176.

ION ABUNDANCE TUNING CRITERIA FOR DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)
Criteria used for GAM 525.2

m/e	RELATIVE ABUNDANCE CRITERIA
51	10 - 80 % of mass 198.
68	Less than 2 % of mass 69.
70	Less than 2 % of mass 69.
127	10 - 80 % of mass 198.
197	Less than 1 % of mass 198.
198	Base peak or >50 % of mass 442
199	5 - 9 % of mass 198.
275	10 - 60 % of mass 198.
365	Greater than 1 % of base peak.
441	Present but less than mass 443.
442	Base peak or 50 % of mass 198.
443	15 - 24 % of mass 442.

Table A - 6
Equations Used for Metals Analysis by ICP-MS
External Standard Quantitation

Parameter	Equation	Units
Water concentration	$(C_x)\,(d)$ where : C_x = concentration of the element being measured from the calibration curve in ug/L d = dilution factor	µg/L
Soil concentration (dry weight)	$\dfrac{(C_x)\,(V_t)\,(d)}{(W_s)}$ where : C_x = concentration of the element being measured from the calibration curve in ug/L W_s = initial dry weight of sample in Kg d = dilution factor V_t = final volume of digestate in liters	mg/Kg

TABLE A-7.
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR WATER SAMPLES

CHEMICAL ANALYSES	MINIMUM VOLUME (mL)*	PLASTIC OR GLASS	PRESERVATION	HOLDING TIME
Acidity	100	P, G	cool, 4°C	14 days
Alkalinity	100	P,G	cool, 4°C	14 days
Ammonia	100	P,G	cool, 4°C sulfuric to pH<2	28 days
BOD 5 day	1000	P,G	cool, 4°C	48 hours
Total Organic Carbon (TOC)	50	P,G	cool, 4°C sulfuric to pH<2	28 days
Chemical Oxygen Demand	50	P,G	cool, 4°C sulfuric to pH<2	28 days
Chloride	100	P,G	none	28 days
Chromium, hexavalent	100	P,G	cool, 4°C	24 hours
Color	50	P,G	cool, 4°C	48 hours
Conductance	100	P,G	cool, 4°C	28 days
Cyanide		P,G	cool, 4°C	14 days
free	100		NaOH to pH>12	
total	500			
total & ammenable	1000			
Fecal Coliform	100	P,G	cool, 4°C	6 hours
Fluoride	100	P	none	28 days
Hardness	100	P,G	none	6 months
Metals (except mercury) (200.7, 200.8, 6010 & 6020)	500	P,G	nitric to pH<2	6 months
Mercury (245.1, 7470)	500	P,G	nitric to pH<2	28 days
Nitrogen		P,G	cool, 4°C	28 days
total Kjeldahl	100		sulfuric to pH<2	
Nitrate	100			
Nitrite	100			
Oil and Grease (413.1)	1000	G only	cool, 4°C sulfuric to pH<2	28 days
Organic Analytes				
Volatiles BTEX, VOC's, TPH/GRO (8020, 8260A, 8260B 8015mod)	40 (3 per/sample)	G only PTFE septa for VOA's	cool, 4°C HCl to pH<2	14 days
EDB, DBCP (8011)	40 (3 per sample)	G only PTFE septa for VOA's	cool, 4°C, 4 drops of 10% Na2S203.	14 days
Semi-volatiles (505)	40 (3 per sample)	G only PTFE septa for VOA's	cool, 4°C, 3 mg of Na2S203.	7 days to extr.
TPH/DRO	1000	G only	cool, 4°C	7 days to extr
Semi-volatiles (8270B)	1000	with PTFE	cool, 4°C	40 days after
Semi-volatiles (525.2)	1000	lined cap	cool, 4°C, 50 mg of Na2SO3	extr.
Pesticides/PCB's (8081)	1000		cool, 4°C	
Herbicides (8150, 8151)	1000		cool, 4°C	
PH (150.1)	100	P,G	none	immediately
Phenolics	100	G only	cool, 4°C sulfuric to pH<2	28 days
Phosphorous				
ortho phosphate	50	P,G	filter on site	48 hours
total phosphate	50	P,G	cool, 4°C	48 hours

TABLE A-7. (cont.)
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR WATER SAMPLES

CHEMICAL ANALYSES	MINIMUM VOLUME (mL)*	PLASTIC OR GLASS	PRESERVATION	HOLDING TIME
Solids		P,G	cool, 4°C	
dissolved	100			48 hours
suspended	250			7 days
total	250			7days
volatile	250			7 days
settleable	1000			48 hours
Sulfate	50	P,G	cool, 4°C	28 days
Sulfide	500	P,G	cool, 4°C 2 mL ZnOAc and NaOH to pH>9	7 days
Sulfite	50	P,G	none	immediately
Total Recoverable Petroleum Hydrocarbons (418.1)	1000	G only with Teflon lined cap	cool, 4°C HCl to pH <2	7 days
Turbidity	100	P,G	cool, 4°C	48 hours

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES AND HOLDING TIMES FOR SOIL SAMPLES

CHEMICAL ANALYSES	MINIMUM CONTAINER SIZE (mL)*	PLASTIC OR GLASS	PRESERVATION	HOLDING TIME
Nutrients/TOC	400	P	cool to 4° C	14 days
Metals (except mercury) (6010 & 6020)	250	P or G	none required	6 months
Mercury	250	P or G	cool to 4° C	28 days
Volatile Organics (SW846 8240, 8260, 8020, 8010)	125	G only with PTFE lined cap	dark, cool to 4° C	14 days
Volatile Organics Low Level (SW846 8260B/5035)	3 x 40 mL	G only with PTFE lined cap	5ml of 20% sodium bisulfate, dark, cool to 4° C	14 days
Volatile Organics High Level (SW846 8260B/5035)	1 X 40 mL	G only with PTFE lined cap	10 mL of purge and trap grade methanol, dark, cool to 4° C	14 days
Volatile Organics percent moisture (SW846 8260B/5035)	1 X 40 mL	G only with PTFE lined cap	dark, cool to 4° C	14 days
Semi-volatile organics, TPH, pesticides, PCB's, etc. (SW846 8270, 8080)	250	G only with PTFE lined cap	dark, cool to 4° C	14 days before extraction, 40 days after extraction
BTEX (SW846 8020)	125	G only with PTFE lined cap	dark, cool to 4° C	14 days
TPH by GC (SW846 8015 modified)	125	G only with PTFE lined cap	dark, cool to 4° C	14 days
TPH by IR (EPA 418.1)	125	G only with PTFE lined cap	dark, cool to 4° C	7 days

** - These are recommended minimum volumes for individual parameters. Some parameters with similar requirements may be combined in a larger container. Please call the laboratory for advice if you wish to combine sampling containers for multiple analyses*

ATTACHMENT D

CHAIN OF CUSTODY FORM, CUSTODY SEAL AND SAMPLE LABELS

CUSTODY SEAL

DATE ______________________

SIGNATURE ______________________

QEC Quality Environmental Containers
800-255-3950 • 304-255-3900

Quality Environmental Containers

PROJECT NAME

SAMPLE ID	SAMPLE DATE	SAMPLE TIME
SAMPLED BY	PRESERVATIVE	
ANALYSIS REQUESTED		___ GRAB ___ COMPOSITE

PROJECT NAME

SAMPLE ID	SAMPLE DATE	SAMPLE TIME
SAMPLED BY	PRESERVATIVE	
ANALYSIS REQUESTED		___ GRAB ___ COMPOSITE

PROJECT NAME

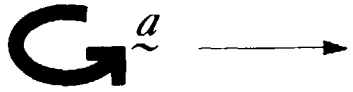
SAMPLE ID	SAMPLE DATE	SAMPLE TIME
SAMPLED BY	PRESERVATIVE	
ANALYSIS REQUESTED		___ GRAB ___ COMPOSITE

PROJECT NAME

SAMPLE ID	SAMPLE DATE	SAMPLE TIME
SAMPLED BY	PRESERVATIVE	
ANALYSIS REQUESTED		___ GRAB ___ COMPOSITE

QEC analytical, Inc

9263 Ravenna Rd Suite A-7
Twinsburg, OH 44087
Phone Number 330 963 6990
Fax Number 330 963 6975



CHAIN OF CUSTODY RECORD

COMPANY NAME AND ADDRESS						NO. OF CON-TAINERS	PROJECT NUMBER AND DESCRIPTION:									
SAMPLER SIGNATURES:							Analysis Requested								VAP	
STA.#	DATE	TIME	COMP.	GRAB.	STATION LOCATION										NOTES	

CHAIN OF CUSTODY SIGNATURES *(Name, Company, Date, Time)*

1. *Relinquished By*: _____
   Received By: _____
3. Relinquished By: _____
   Received By: _____
2. Relinquished By: _____
   Received By: _____
4. Submitted to Laboratory By: _____
   Received for Laboratory By: _____

CHAIN OF CUSTODY

ENTACT

1360 N. Wood Dale Rd. Suite A
Wood Dale, Illinois 60191
Ph. 630/616-2100 Fax 630/616-9203

Sampler:_____ Job #:_____

ENTACT Contact:_____ Date:_____

Turnaround Time Requested
24 Hour[ ] 48 Hour[ ] 3 Day[ ] Normal[ ] Other[ ]_____

Sample No.	Matrix	Composite or Grab	Description/Remarks	Preservative	Analysis

Samples Relinquished By: _____ Date

Samples Received By: _____ Date

Samples Relinquished By: _____ Date

Samples Received By: _____ Date

Samples Relinquished By: _____ Date

ANALYSIS

A=_____ F=_____

B=_____ G=_____

C=_____ H=_____

D=_____ I=_____

E=_____ J=_____

Distribution:

Original - To Customer w/ Final Report
2nd Copy - To Job File
3rd Copy - To Lab

Condition of Sample Upon Receipt:

Bottles Intact? Yes / No	Volatiles Free of Head ace? Yes / No	COC Seals Presen d Intact? Yes / No

AIR MONITORING LOG

ENTACT
1360 N. Wood Dale Rd Suite A
Wood Dale, Illinois 60191
Ph. 630/616-2100 Fax 630/616-9203

Sampler:_____ Job #:_____

ENTACT Contact:_____ Date:_____

Sample No.	Instrument I.D.	Time	Flow Rate	Volume	Type of Sample	Analysis

NOTES AND CALCULATIONS

Distribution:

Original - To Customer w/ Final Report
2nd Copy - To Job File
3rd Copy - To Lab

APPENDIX E

LABORATORY ACCREDITATIONS AND CERTIFICATIONS

OhioEPA

OHIO E.P.A.

DEC 21 2000

ENTERED DIRECTOR'S JOURNAL

Ohio Environmental Protection Agency
Division of Emergency and Remedial Response
Voluntary Action Program

Under the authority of Ohio Revised Code Section 3746.04(B)(6) and Ohio Administrative Code Rule 3745-300-04

Certifies

GEOANALYTICAL, INC.
9263 RAVENNA ROAD, SUITE A-7
TWINSBURG, OH 44087

as a

Certified Laboratory
(Number CL0008)

for the following analytes, parameter groups, and methods

Aluminum/200.8,6010A,6020	Cobalt/200.8,6010A,6020	Potassium/6010A	Volatile Organic Compounds/8020A,8260A,8260B,
Antimony/200.8,6010A,6020	Copper/200.8,6010A,6020	Selenium/200.8,6010A	Semi-Volatile Organic Compounds/8270B
Arsenic/200.8,6010A,6020	Iron/6010A	Silver/200.8,6010A,6020	Organochlorine Pesticides/8081
Barium/200.8,6010A,6020	Lead/200.8,6010A,6020	Sodium/6010A	Polychlorinated Biphenyls/8081
Beryllium/200.8,6010A,6020	Lithium/6010A	Thallium/200.8,6010A,6020	Polynuclear Aromatic Hydrocarbons/8100
Cadmium/200.8,6010A,6020	Manganese/200.8,6010A,6020	Vanadium/200.8,6010A	1,2-Dibromoethane and 1,2-Dibromo/3-Chloropropane/8011*
Calcium/6010A	Mercury/7470A,7471A	Zinc/200.8,6010A,6020	Total Petroleum Hydrocarbons, Diesel Range Organics/418.1, 8015A-Modified
Chromium/200.8,6010A,6020	Nickel/200.8,6010A,6020	n-Hexane/8260A-Modified**	Total Petroleum Hydrocarbons, Gasoline Range Organics/8015A-Modified

*Water **In accordance with all documentation submitted pursuant to OAC Rule 3745-300-04(D) and approved by the VAP.

DEC 21 2000
Date of Certification

Director, Ohio Environmental Protection Agency

NOV 22 2002
Date of Expiration

Manager, Voluntary Action Program

SCOPE, LIMITATION, OBLIGATIONS AND RESPONSIBILITIES OF CERTIFICATION ON REVERSE SIDE

Commonwealth of Kentucky

Department for Environmental Protection

Division of Environmental Services

Certificate of Laboratory Certification

for the Chemical Analysis of Drinking Water

in accordance with 401 KAR Chapter 8, issued to:

Geo Analytical, Inc.
9263 Ravenna Road, Suite A-7
Twinsburg, Ohio 44087

for the analytes listed on the most current certified parameter list.

Scott Byrum

Steven L. Wheatley

Certification Officers

Laboratory ID # 90081

Expires December 31, 2001

APPENDIX E

TREATABILITY STUDY REPORT

1997 FINAL TREATABILITY STUDY
FOR
THE MASTER METALS, INC. SITE
CLEVELAND, OHIO

PREPARED BY
ENTACT, Inc.

September 2, 1997

1.0 INTRODUCTION

ENTACT, Inc. conducted a treatability study in 1997 for the Master Metals Technical Committee (MMTC) to obtain information from which to design a stabilization/solidificationprocess that is best suited for treating lead, cadmium, and arsenic contaminated feedstock and soils at the Master Metals Site in Cleveland, Ohio. Based upon information collected during time-critical removal activities, materials present on site contain as much as 155,000 ppm lead. The leachability of the lead in these materials is also very high with TCLP tests performed under the direction of ENTACT showing some levels above 2,000 mg/L. The stabilization/solidificationprocess must reduce the leachability of lead, cadmium, and arsenic to nonhazardous levels. ENTACT performed a bench scale study of treatment alternatives in a manner that replicates the performance of the additives in actual field implementation. The results that are obtained in the laboratory study can be expected for materials that are stabilized in the field.

In addition to rendering the materials nonhazardous, ENTACT directed the treatability study to find the optimum blend of additives which effectively rendered the soils nonhazardous. An important consideration in determining the optimum blend was finding the minimum amount of additive that would successfully treat the on site materials. Optimization of the additive quantities in the blend will reduce the total amount of additives needed for the project. This minimization of additives will also help tqprevent a volume increase in the treated material. The reduction in volume of treated material will reduce handling costs as well as disposal and containment costs. ENTACT has developed a patented blend of additives that significantly reduces the leachability of lead and other heavy metals and also minimizes the amount of treatment reagent added to the material. The additives that ENTACT utilizes are very effective. Materials treated with these additives become stable and resist leaching. The additives have a bufferingcapacity which keeps the treated matrix in a pH range that provides long term durability and reduces the leachability of heavy metal components. These patented additives have been used successfully for treating over 1,000,000 tons of contaminated soil, slag, battery components, and other debris.

2.0 AS-RECEIVED MATERIAL ANALYSIS

In July and August 1997, ENTACT collected twelve sample containers containing representative samples of materials from the Master Metals Site. Samples were collected from the on-site soils, drummed waste and feedstock materials. The matrices were collected according to waste streams found on site in the following classifications:

- gross contamination removed from the concrete slab during remediation activities;
- refractory brick and debris drummed on site;
- soil contained in roll off boxes;
- material originating from furnaces and ball mills;
- white lead waste from drums and supersacks in feedstock area;
- gray lead powder waste from supersacks in feedstock area;
- leaded glass in pallet containers and roll off boxes;
- large gray waste pile near furnace building;
- **excavated soils from western and southern portions of site;**
- sample material received on site prior to refining;
- by-products of glazing process (roll off boxes); and
- drummed solid waste material

These materials were taken to National Environmental Testing Laboratories (NET), of Bartlett, Illinois, for treatability analysis.

Prior to as-received sample analysis, it was determined that materials, being of a heterogeneous nature, would be analyzed as separate waste streams and treated as such. Tests were conducted to determine the total metals concentration and TCLP metals on the one composite sample. These tests established baseline conditions against which treatment additives were evaluated. The results are provided in Table 1.

Total metal concentrations for lead ranged from 12,000 mg/kg to 155,000 mg/kg. As shown in Table 1, all materials exhibited the toxicity characteristic for lead. Toxicity characteristic concentrations for lead ranged from 42.8 mg/L to 2,180 mg/L. Nine of the thiteen waste streams exhibited the toxicity characteristic for cadmium. Toxicity characteristic concentrations for cadmium ranged from less than 0.1 mg/L to 75.3 mg/L. Three of the thirteen waste streams exhibited the toxicity characteristic for arsenic.

Toxicity characteristic concentrations for arsenic ranged from 0.023 mg/L to 15.1 mg/L.

3.0 LEAD STABILIZATION

3.1 Behavior of Lead in Materials

Lead is the primary contaminant of concern at the Master Metals Site due to its ubiquity in feedstock material, waste piles, and surficial soils (see Table 1). Elemental lead (Pb), lead sulfate ($PbSO_4$), lead oxide (PbO), and lead dioxide (PbO_2) are the predominant species found. Sites with carbonate soils generally contain lead carbonate ($PbCO_3$), hydrocerussite ($Pb_3(CO_3)_2(OH)_2$), or lead hillite ($Pb_4SO_4(CO_3)_2(OH)_2$). Other heavy metals, such as antimony, arsenic, cadmium, and copper are sometimes present, but normally at relatively low concentrations (Royer, 1992).

Lead is generally not very mobile in the environment, and tends to remain relatively close to its point of initial deposition. Generally, soils tend to retain lead in the upper few centimeters. The capacity of soil to adsorb lead increases with increasing pH,cation exchange capacity, organic carbon, soil/waterEh (redox potential), and phosphate levels. Lead exhibits a high degree of adsorption on clay-rich soil. Lead compounds can also be adsorbed onto hydrous oxides of iron and manganese and thus be immobilized in salts containing two or more cations (Royer, 1992).

In order for chemical fixation/stabilization to be successful, the various forms of lead salts, especially lead oxide, need to be converted to compounds that are particularly insoluble under the normal pH range. Lead is capable of forming the following three low solubility orthophosphate salts:

$Pb_3(PO_4)_2$,
Pb_2HPO_4 , and
$Pb(H_2PO_4)_2$.

3.2 Treatment Technology Description

The stabilization process, sometimes referred to as immobilization or fixation, uses additives to chemically immobilize the hazardous constituents of a contaminated material by combining the additives and lead-bearing matrix within a mixing device. Additive reagents for use in the stabilization of lead contaminated materials include Portland Cement, calcium oxide, calcium carbonate, fly ash, and proprietary additives (EPA, 1989 and Conner, 1990). Other investigators have documented successful stabilization of lead using combinations of the following compounds: magnesium oxide, magnesium hydroxide, reactive calcium carbonates, reactive magnesium carbonates, and boric acid.

ENTACT has developed a proprietary list of additives for stabilizing waste containing lead and other heavy metals including phosphoric acid, monocalcium phosphate (TSP), monoammonium phosphate, and diammonium phosphate either alone or in combination with Portland Cement.

The listed ENTACT patented compounds provide the necessary environment for successful lead stabilization. The first component is a phosphate ion that reacts with metals such as lead to form a salt which is insoluble under normal environmental conditions. The second component is the phosphoric acid buffer system that provides stability to the treated waste mixture under minor environmental changes.

The stabilization process and ENTACT patented additives provide the necessary components for successful stabilization of lead contaminated soil and debris–small and consistent particle size, a phosphate ion, a buffering system, and thorough mixing.

4.0 ADDITIVE DESIGN AND SELECTION

All thirteen separate samples, representing the waste streams shown in Table 1, were prepared for treatability testing. The percentages of the additive components used in the blend were varied for several trials. The additive blend varied from two percent to ten percent based upon the matrix characteristics. This relatively wide range of additive mixtures is due in part to the heterogeneous nature of materials present on site. After receiving results from this initial treatability study it was determined that some waste streams required the addition of cement along with the phosphate. These materials were treated with blends of 5 percent phosphate-5 percent cement and 5 percent phosphate-10 percent cement.

ENTACT's combination of additives is a proprietary blend of chemical agents which greatly reduces the leachability of metals in the stabilized matrix. The leachability of the metals in the treated matrix will be reduced to nonhazardous levels with an appropriate blend of additives. The leachability of treated materials indicates the effectiveness of the various blends. TCLP tests measured the leachability of lead, cadmium, and arsenic from the matrix following treatment.

4.1 Selection of Additive Blend

Each of the samples was analyzed separately with an initial additive blend mixture of two percent. This mixture was increased in increments of two (i.e., 4%, 6%, etc.) or a blend of cement/phosphate was utilized until the matrix was rendered nonhazardous. The samples containing the selected blends were tested for TCLP metals. The results of these tests are provided in Table 2. As shown in Table 2, all but two of the waste streams were successfully treated to non-hazardous characteristic levels. The determination was made based on the developing trend for SDY-05 and SDY-10 that the material could not be treated without a substantial addition of additive. Therefore, it was determined that these two waste

streams would not be treated on site. Instead, these materials will be consolidated into roll-off boxes and disposed of as hazardous waste.

Eight of the waste streams were treated successfully with a phosphate treatability of 4-8 percent by wieght. Of these, the excavated soils were successfully treated with a phosphate treatability of 4-6 percent. Only three or four waste streams required a treatability blend of phosphate/cement.

ENTACT has used similar additive blends (the same constituents in different proportions) at numerous heavy metal contaminated sites. ENTACT additives offer a very high degree of consistency and reliability resulting in uniform performance characteristics.

5.0 CONCLUSIONS

ENTACT's selected blends of additives were successful in stabilizing the lead contaminated materials in eleven of the thirteen waste streams present at the Master Metals Site. The average TCLP level for lead was reduced from approximately 922 mg/L, to about 3 mg/L, a reduction of more than 99.6%. A similar reduction in TCLP levels for cadmium (98.1%) and arsenic developed from the addition of the cement to the treatability samples. Based upon total metals results, materials on site with like lead concentrations may be mixed prior to treatment to consolidate waste streams and provide effective processing of the waste materials and treatability reagents.

Two blends of additive will be utilized to treat contaminated material at the Master Metals Site - phosphate blend and phosphate/cement blend. The chosen blends of additives met all requirements of the treatability study. The blends result in a minimal addition of material to the contaminated waste stream which will help prevent a volume increase after compaction in the final disposal location. The durability of the final remedy is assured over the long term as a result of the pH buffering capacity of the additives. The ENTACT blend offers better performance for the requirements of land disposal of contaminated materials as compared with other additives.

6.0 REFERENCES

1. United States Environmental Protection Agency, August 1991, "Handbook Stabilization Technologies for RCRA Corrective Actions," (EPA/625/6-91/026), Office of Research and Development, Washington, DC.

2. Royer, Michael D., Ari Selvakumar and Roger Gaire. "Control Technologies for Remediation of Contaminated Soil and Waste Deposits at Superfund Lead Battery Recycling Sites," J Air Waste Management Association, Volume 42, Number 7, pp. 970-980 (1992)

3. Conner, Jessie R., Chemical Fixation and Solidification of Hazardous Waste, Van Nostrend Reinhold (1990).

Table 1
Analysis of Material Samples As-Received

Sample ID	Description	Total Metals Concentration (mg/kg)			TCLP Metals Result (mg/L)		
		Pb	Cd	As	Pb	Cd	As
SDY-01	gross surface contamination	89,000	1,500	1,300	1,390	55.5	<2.0
SDY-02	refractory brick	80,000	188	<500	2,180	1.26	15.1
SDY-03	soil-roll off	56,000	89	154	52.0	-	-
SDY-04	furnace/ball mill materials	86,000	3,400	545	42.8	75.3	-
SDY-05	gray powder	75,000	3,440	<500	782	51.2	5.15
SDY-06	white lead powder	70,000	<25	<500	1,210	0.045	8.59
SDY-07	leaded glass	12,000	-	-	177	<0.010	<2.0
SDY-08	dark gray waste pile	48,000	110	180	840	1.38	0.023
SDY-09	on site soil-excavated	17,000	61	220	1,400	3.54	0.0899
SDY-09-2	on site soil-excavated	77,000	68	230	508	2.72	< 2.0
SDY-10	refining samples	84,000	18	430	1,090	< 0.10	< 2.0
SDY-11	glazing by-product	155,000	320	380	1,900	3.86	<2.0
SDY-12	drummed solid waste material	100,000	630	750	620	6.89	<2.0

Table 2

Analysis of Selected Blends

Sample ID	Treatment	Description	Total Metals (ug/g) Pb	Cd	As	TCLP Metals (mg/L) Pb	Cd	As	soil pH
SDY-01		**gross surface contamination**	96,000	1,500	1,300	1,390	55.5	<2.0	6.99
	2%	treatment reagent added				1,340	-	-	6.9
	4%	treatment reagent added				577	-	-	6.7
	6%	treatment reagent added				194	-	-	6
	8%	treatment reagent added				114	-	-	-
	5%-5%	treatment blend added				9.36	2.44	<2.0	
	10%-5%	treatment blend added							
SDY-02		**refractory brick**	80,000	188	< 500	2,180	1.26	15.1	7.1
	2%	treatment reagent added				1,050	-	-	6.41
	4%	treatment reagent added				966	-	-	3.92
	6%	treatment reagent added				3.57	-	-	4.32
SDY-03		**roll off boxes of soil/gravel**	56,000	89	154	52	-	-	7.66
	2%	treatment reagent added				1.51	-	-	5.65
	4%	treatment reagent added				1.08	-	-	6.42
	6%	treatment reagent added				0.509	-	-	4.92
SDY-04		**furnace and ball mill material**	86,000	-	-	42.8	75.3	-	7.21
	2%	treatment reagent added				61.9	27.6	-	6.44
	4%	treatment reagent added				49.2	28.8	-	5.22
	6%	treatment reagent added				4.03	10	-	5.71
SDY-04-2		furnace and ball mill material	65,000	3,400	-	30.8	130	-	6.44
	8%	treatment reagent added				5.44	31.7	-	-

Table 2

Analysis of Selected Blends

Sample ID	Treatment	Description	Total Metals (ug/g) Pb	Cd	As	TCLP Metals (mg/L) Pb	Cd	As	soil pH
	15%	treatment reagent added				2.01	1.97	1.59	4.57
	10%-5%	treatment blend added				< 0.80	< 0.050	< 2.0	10.01
SDY-05		**gray powder (supersacks)**	75,000	-	-	782	51.2	5.15	6.55
	2%	treatment reagent added				557	-	-	6.49
	4%	treatment reagent added				537	-	-	6.42
	6%	treatment reagent added				530	-	-	6.45
SDY-06		**white powder (drums & supersacks**	70,000	< 25	< 500	1,210	0.045	8.59	11.38
	2%	treatment reagent added				27.7	-	-	12
	4%	treatment reagent added				5.4	-	-	11.57
	6%	treatment reagent added				1.42	-	-	9.96
SDY-07		**lead glass material**	12,000	-	-	1.26	<0.10	<2.0	5.67
	2%	treatment reagent added				16.2	-	-	3.25
	4%	treatment reagent added				< 0.800	-	-	2.85
	6%	treatment reagent added				3.28	-	-	2.95
	8%	treatment reagent added				4.27	-	-	-
SDY-08		**dark gray waste pile**	48,000	110	180	840	1.38	0.0226	6.78
	2%	treatment reagent added				900	4.09	-	-
	4%	treatment reagent added				4.34	0.286	-	-
	6%	treatment reagent added				1.42	0.172	-	-

Table 2

Analysis of Selected Blends

Sample ID	Treatment	Description	Total Metals (ug/g) Pb	Cd	As	TCLP Metals (mg/L) Pb	Cd	As	soil pH
SDY-09		**excavated soil from on site**	17,000	61	220	1400	3.54	0.0899	6.17
	4%	treatment reagent added				877	1.47	-	-
	6%	treatment reagent added				408	1.32	-	-
	8%	treatment reagent added				45.8	1.11	-	-
SDY-09-2		excavated soil from on site	77,000	68	230	508	2.72	< 2.0	5.22
	2%	treatment reagent added				18.9	-	-	5.53
	4%	treatment reagent added				1.59	-	-	5 64
	6%	treatment reagent added				4.06	-	-	5.02
SDY-10		**composite of 2 gal sample buckets**	84,000	18	430	1,090	< 0.10	< 2.0	8.92
	6%	treatment reagent added				961	-	-	7.68
	8%	treatment reagent added				556	-	-	6.51
	10%	treatment reagent added				356	-	-	6.17
SDY-11		**roll off boxes of glazing by-product**	155,000	320	380	1,900	3.86	<2.0	7.04
	10%	treatment reagent added				122	2.29	< 2.0	5.06
	5%-5%	treatment blend added				92	1.95	<2.0	8.84
	10%-5%	treatment blend added				9.64	<0 050	<2.0	11.14
SDY-12		**solid drummed material (dross, etc)**	100,000	630	750	620	6.89	<2.0	6.19
	10%	treatment reagent added				293	4.86	<2.0	6.92
	5%-5%	treatment blend added				110	0.19	<2.0	7.7
	10%-5%	treatment blended added				0.125	0.022	<2.0	9.15

APPENDIX F

FINAL EROSION CONTROL PLAN

Master Metals, Inc. Site
EROSION CONTROL PLAN

SITE DESCRIPTION

[redacted]	Master Metals, Inc. Site Cuyahoga County Cleveland, Ohio	[redacted]	

The goal of the Mater Metals, Inc. (MMI) Erosion Control Plan is to implement the baseline best management practices (BMPs) for controlling surface run-off and sediment erosion during construction activities. The Construction Project Manager will ensure these practices are being followed, including completion of inspection logs and assisting with sampling activities. The ECP will be updated when the nature of on-site activities changes substantially, or information indicates a possible problem in data collection.

[redacted]

The team for the construction activities at the MMI Site is:

Leader: Bob Ainslie **Title:** ENTACT Field Project Manager
Stormwater Responsibilities: Coordinate preparation and implementation of plan; coordinate employee-training programs; coordinate record keeping; and ensure inspections are performed.

Member: Joe Cronk **Title:** On-Site QA/QC Officer
Stormwater Responsibilities: Coordination of ENTACT plans with master plans, coordinate with the Site-wide Stormwater Inspector and the Site-wide Stormwater Administrator. Note any on-site changes that may affect plan; conduct inspections. Has reviewed the ESCP and is familiar with the following: the location and type of control measures, the construction requirements for the control measures, the maintenance procedures for each of the control measures, spill prevention and cleanup measures, and inspection and maintenance record keeping requirements. Mr. Cronk has preformed similar operations and been responsible for these functions at projects of this size and larger.

[redacted]

The construction activities at the MMI Site are being conducted pursuant to a Administrative Order of Consent (AOC) agreed to in Spring 2002 between the Potentially Responsible Parties (PRPs) and United States government and filed under Sections 106(a), 107 and 122 of the Comprehensive Environmental Response, Compensation and Liability Act and the Statement of Work (SOW).

The SOW required excavation of approximately 1,800 to 3,600 cubic yards of lead-impacted soils along the perimeter of the site for on-site stabilization to render the soils nonhazardous. Soils will be excavated until the risk-based remediation goal of 1,000 mg/Kg is reached or until historic slag is encountered, whichever comes first

The treated material and the stockpiled stabilized soils from the Holmden Avenue removal action not used to fill depressions beneath a constructed asphalt cover system will be transported off-site for disposal at the approved Subtitle D landfill . All excavation areas and all areas disturbed by construction will be backfilled with suitable clean fill that meets the performance standards outlined in the FSAP, graded to provide positive drainage, and to

control any additional ponding of water that may occur during implementation of the remedy.

Any necessary drainage ditches, drainage swales, and erosion control methods will be provided to prevent surface runoff during construction from eroding the final grade or flowing off-site

The scope of work for the remedial action includes the following major construction activities:

- Clear and grub areas requiring excavation of all trees and dispose of off-site.
- Excavation of lead-impacted soils on-site that are not under concrete or the asphalt cover, nor addressed during that Phase I TCR, and off-site soils along the western, eastern and southern perimeter of the MMI facility, that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first. XRF screening technology will be used to guide depth of the excavations during removal.
- Backfill all excavated areas determined to have met the RBRG or have reached historic slag fill, with clean imported fill material that has been approved for use based on analytical results.
- Stabilization of excavated on-site and off-site impacted soils to achieve a Toxicity Characteristic Leaching Potential (TCLP) value of less than 5.0 mg/L lead that will meet the applicable LDRs for contaminated soils (<7.5 mg/L) and render the material nonhazardous (<5.0 mg/L).
- Off-site disposal of all treated soils, including stockpiled soils from the Holmden Properties Removal Action not used to fill depression beneath the asphalt cover.
- Construction of a minimum 4-inch thick asphalt cover over the deteriorated concrete in the southern portion of the site.
- Recondition existing concrete surfaces not under the asphalt cover by sealing any significant cracks or deteriorated areas that extend through the concrete surface, followed by encapsulation of the concrete surface, in accordance with the SOW and approved design plan.
- Removal of any existing solid waste including Investigative Derived Waste (IDW) from previous or current removal actions.
- Monitoring air, ground water and sediment monitoring during the RA.
- Installation of dust and storm water controls during the implementation of the RA;
- Site restoration;
- Initiation of Operation and Maintenance Plan ns at the site.

A project schedule of the major activities to be conducted at the site is provided in Figure 6-1 in the RA Workplan.

	The MMI Site is an approximately 4.3-acre lot in an industrial area of Cleveland. The site is flat, predominantly covered with concrete. All former buildings, except the roundhouse and adjoining office buildings have been razed. Remaining areas requiring excavation are located along the site perimeter. An asphalt cover system will be constructed in the southern half of the property in accordance to the design specifications. See Figure 4-1 of the RD/RA Workplan showing topography and proposed cover system location.
Site Drainage System	Existing on-site catch basins connecting to the municipal sewer system have been determined to be functional. The locations are presented in the design specifications. The sewers located in the south-central portion of the site is the access for a 36-inch diameter line flowing east to west. Two eight-inch clay tiles connect to the main line of this manhole. One flows into the 36-inch line from the north and the other flows into the 36-inch line from the southwestern direction. Surface water drainage is sloped toward the existing on-site sewers, with some uncontrolled stormwater run-off collecting in a low-lying area located in the central portion of the site. In accordance to the design specifications, grading of the proposed asphalt cover will direct surface run-off on property to the existing catch basins.
Areas Potentially Receiving Surface Water Runoff from Site	The drainage on-site is directed toward stormwater catchment basins. All sumps that were present on site were cleaned out as part of the Phase 1 TCR and will be backfilled and covered as part of the RA. The B&O Railroad embankment along the western boundary of the site and West 3rd Street to the south and east provide manmade barriers to off-site migration.
Site Map	All pertinent surrounding features, including water resources, topography, and drainage patterns and facilities are illustrated in Figure 1-2 of the RD/RA Workplan
Soil Types	The soils underlying the site consist primarily of fill materials (i.e., cinders, slag, sand) and reworked native silt and clay soils. The native soils are glacial till consisting of silt, clay and sand. On-site boring logs indicate that a sand or a sandy clay fill material with some construction debris ranges in depth between two to four feet and overlies a historic slag and cinder fill.
Name of Receiving Waters	There are no existing surface water bodies on-site. The nearest surface water body is the Cuyahoga River, located 1,300 feet east of the site. It is unlikely that run-off from the site could reach the river due to intervening natural and manmade barriers in this heavy industrialized area of the city.

CONTROLS

Erosion and Sediment Controls

Short and Long Term Goals and Criteria

Erosion and sediment controls consist of measures to divert upslope water around disturbed areas, to maintain all site drainage within the excavation area, and to remove sediment from storm water before it reaches the storm sewer system. During all phases of construction, care will be taken to minimize the disturbed area, and to stabilize disturbed areas as soon as possible. The control measures to be implemented as part of this ECP include:

- Berms surrounding the stockpile area; and
- Stabilized construction entrances.

Ground to be disturbed in the MMI site will drain back into the excavations since the streets and adjoining sidewalks are constructed above grade. ENTACT will limit the time of exposure of disturbed areas by expedited construction schedule and installation of temporary vegetation 14 days after completion of the project. If further construction activities are scheduled for the site within 21 days after project completion, there will be no installation of temporary vegetation. ENTACT is responsible for implementation of these controls.

Design of Control Measures

The erosion and sediment controls are designed to retain sediment on-site to the extent practicable during excavation activities. The controls will be installed according to manufacturer's instructions and according to good engineering practices.

As required by Ohio Administrative Code 1501: 15-1-04(A)(12), all temporary erosion and sediment control measures will be dismantled within thirty (30) days after final site stabilization is achieved or after the temporary practices are no longer needed, unless otherwise authorized by the approving agency. Trapped sediment will be permanently stabilized to prevent further erosion.

As required by Ohio Administrative Code 1501: 15-1-04(A)(11), a permanent vegetative cover must be on denuded areas not otherwise permanently stabilized and will not be established until ground cover provides adequate cover and is mature enough to control soil erosion satisfactorily and survives adverse weather conditions.

CONTROLS

Sediment Management	Off-site accumulations of sediment will be removed weekly to minimize off-site impacts.
Timing of Control Measures	The treatment containment area will be constructed prior to clearing or grading of any portion of the site. The 12-inch to 18-inch berms surrounding the stockpile area will be constructed before any stockpiling activities commence. Areas where construction activity ceases for more than 21 days will be stabilized with temporary seed and mulch within 14 days of the last disturbance. Once construction activity ceases permanently in an area, that area will be stabilized with permanent seed and mulch. After the entire site is stabilized, the accumulated sediment will be removed and silt fences will be removed.
Waste Disposal	**Waste Materials:** All non-hazardous construction debris and general office trash will be disposed in a roll-off or dumpster placed in the support zone. Trash receptacles will be placed in the storage trailer and office trailer for the collection of non-hazardous trash and debris. Spent personal protective equipment will not be disposed with non-hazardous trash. **Hazardous Materials:** Previous investigation information has identified two categories of materials that will be excavated during remediation activities: topsoil, and impacted fill material. Each material type will be staged in designated storage areas. Stockpiled areas will be covered to prevent erosion and storm water runoff. A 12-inch to 18-inch berm will be constructed surrounding the stockpile area. Collected storm water will be sampled, treated, (if necessary), and discharged into storm drains or used in dust suppression.

Stabilization Practices

Site Stabilization

Within 14 days of completion of excavation, backfilling and final grading activities in each excavation area of the site, the site will be stabilized. All sediment control devices located at storm inlets will remain in place and will be maintained. If other construction activities will commence sooner than 21 days after final grading in an area, then no stabilization will be completed.

The following records will be maintained and attached to the ESCP:

- the dates when major grading activities occur;
- the dates when construction activities temporarily or permanently cease on a portion of the site; and,
- the dates when stabilization measures are initiated.

Storm Water Management Strategy

Surface Conditions	The site is predominately covered with concrete. Areas not covered with concrete requiring excavation are covered with grass. After the remedy is completed, excavated areas will be re-graded and re-vegetated. This action will cause the peak rates of run-off before development to be less after the construction is completed, as required pursuant to Ohio Administrative Code 1501:15-1-05
Post-Construction Pollution Prevention	Designs for future construction and associated stormwater control structures have not been finalized. Operation and maintenance of these controls will be transferred to the Site-wide Stormwater Administrator upon completion of stabilization activities.

Other Controls

No solid materials, including building materials, will be discharged directly to waters of the United States. Therefore, no permit issued under section 404 of the CWA is required.

Off - Site Vehicle Tracking	A stabilized construction entrance has been provided to help reduce vehicle tracking of sediments. The entrance will be swept, as needed, to remove any excess mud, dirt or rock tracked from the site. All vehicles hauling waste materials from the project site will be tarped.
Local, Tribal, and State Compliance	Off-site discharge will be coordinated with the Northeast Ohio Regional Sewer District (NEORSD).
Materials Stored On-site	Hydraulic oils, motor oils and lubricants will be stored in the on-site equipment storage trailer. Quantities of these items should not exceed 10 gallons. If larger quantities of these items are required to be on-hand, ENTACT will review the storage and containment of those items at such time.
Spill Prevention and Response	Pollution prevention measures will include implementation of best management practices (BMPs). If a reportable quantity of oil or hazardous material release is discovered, ENTACT will notify the National Response Center 800/424-8802 immediately. The Ohio Environmental Protection Agency (OEPA) will be notified verbally within 24 hours and in writing within 14 days. Complete emergency response and spill cleanup procedures are detail in the Site Specific Health & Safety Plan. The ESCP will also be modified to include: - the date of the release; - circumstances leading to the release; and - steps taken to prevent reoccurrence of the release.
Pollution Sources from Areas Other Than Construction	The site is inactive so there are no other pollutant sources in the form of other industrial operations.
Updating the ECP	This ECP will be updated as necessary to remain consistent with site changes and/or regulation changes that affect stormwater management at the site.

MAINTENANCE / INSPECTION PROCEDURES

Erosion and Sediment Control Inspection and Maintenance Procedures

Maintenance of the storm water controls has been identified as a major part of effective erosion and sediment programs. ENTACT will perform inspections at least once every 14 calendar days and within 24 hours of any storm event of greater than 0.5 inches. The Field Project Manager will perform inspections and the inspection reports will be maintained. The Field Project Manager will inspect disturbed areas and areas used for storage of materials that are exposed to precipitation for evidence of, or the potential for, pollutants to enter the runoff from the site. Erosion and sediment controls will be inspected to ensure they are functioning properly and that they are positioned adequately for the control of runoff and sediment. Storm water inlets will be inspected for evidence of sediment accumulation or flow restriction. Locations where vehicles enter or exit the site will be inspected for evidence of offsite sediment tracking. Based on the results of the inspection, the pollution prevention measures will be revised as soon as possible, not less than 7 days, after inadequacies are revealed. The inspection reports will be retained as part of the Storm Water Pollution Prevention Plan for at least three years after the date of inspection.

NON-STORMWATER DISCHARGES

ENTACT will control transient dust through active use of water-based control methods. These include high-pressure water misting units placed in and around excavation work areas. The misting units will be used to control fugitive emissions in active work areas.

EROSION AND SEDIMENT CONTROL PLAN INSPECTION AND MAINTENANCE RECORD

1. Inspections to be preformed bi-weekly & within 24 hours of a rainfall of 0.50" or greater;
2. Inspect storm water drainage areas for evidence of pollutants entering the drainage systems;
3. Evaluate the effectiveness of controls and best management practices (good housekeeping activities, preventive maintenance practices, etc.)
4. Observe structural measures, sediment controls, vegetative cover and other storm water best management practices to ensure proper function or proper condition;
5. Revise the plan as needed within 1 week of inspection and implement any necessary physical changes within 1 week of inspection;

OBSERVATIONS:

Inspected by: _____ Date: _____

APPENDIX G

FINAL COMMUNITY RELATIONS PLAN

1.0 COMMUNITY RELATIONS PLAN

1.1 Overview of Community Relations Plan

This Community Relations Plan (CRP) is prepared for the Master Metals, Inc. (MMI) Site in Cleveland, Ohio. The CRP outlines community relation activities to be conducted during the implementation of the Removal Action and during the term of the Administrative Order of Consent (AOC) between the United States Environmental Protection Agency and the MMI PRP Group. These USEPA will follow the *June 1997 Final Revised Community Involvement Plan* for the MMI site.

The Community Relations Plan has been prepared to aid the MMI PRP Group and U.S. EPA in developing a community relations program tailored to the needs of the citizens of Cleveland, Ohio. The MMI PRP Group and U.S. EPA will conduct community relation activities to ensure that the local public is well informed about the progress of any actions taken under the AOC. This plan contains the following sections.

- Site Description
- Description of the RA
- Highlights of Program
- Documents Available for Public Review
- List of Contacts
- Information Repository

1.2 Site Location and Description

The MMI Superfund Site (the "Site') covered under the AOC includes the former MMI lead facility (the "Facility") located at 2850 West Third Street, Cleveland, Cuyahoga County, Ohio and an on-site stockpile of treated lead-impacted soils from a 1997 removal action of contaminated soils at 1157, 1159 and 1167 Holmden Avenue (the "Holmden Properties") where lead-impacted material from Master Metals was deposited as fill (USEPA, 1999). The site is situated in Township 7 North, Range 12 West, Section 17, ¼ NE, ¼ SW, ¼ SW , with coordinates obtained from the Facility Index System (FINDS) listed as 41 degrees, 28 minutes, 26 seconds latitude and -81 degrees, 40 minutes, 31 seconds longitude.. The site location is illustrated in Figure 1.

The Master Metals Inc. (MMI) property is a triangular-shaped parcel encompassing approximately 4.3 acres in the "flats" area of downtown Cleveland, a heavily industrialized sector of the city. The site is bordered on west by rail yards owned by the Baltimore & Ohio (B&O) Railroad, the east by West Third Street and B&O railroad tracks, and on the south by a dead-end road and an abandoned industrial property. LTV Steel owns the property to the south and north. The Cuyahoga River is located approximately 1,250 feet east of the facility and flows north toward Lake Erie (ENTACT, 1999). An athletic field and playground are situated approximately 1,000 feet to the west. The nearest residential property to the former facility is approximately 2,000 feet to the northwest (USEPA, 1999).

Major site features, prior to a 1997-1998 time-critical removal (TCR) action, included an office building, a secondary lead smelting furnace building, two large brick baghouses, the roundhouse building, storage buildings, material storage bins and boxes, and an above-ground storage tank farm (ENTACT, 1998). All buildings, except for the roundhouse and the attached office building in the northern corner of the property, have been razed as part of the Phase I TCR (ENTACT, 1998) and all remaining feedstock and debris materials were decontaminated and/or treated and disposed of off-site as either special waste or as hazardous waste (ENTACT, 1998). The MMI facility property is currently vacant with the exception of the roundhouse, and the majority of the land surface covered with concrete or asphalt except along the site boundaries. Current site features are illustrated in Figure 2.

1.3 Site History

The facility was constructed in 1932 on slag fill by National Lead Industries, Inc. (NL) who owned and operated the facility as a secondary lead smelter, producing lead alloys from lead-bearing dross and scrap materials. NL Industries also engaged in battery cracking operations at this facility. In 1979, the facility was purchased from NL Industries by Master Metals who continued to run secondary lead smelter operations (USEPA, 2001a).

As part of their operations, the Master Metals facility received lead-bearing materials classified and regulated under Resource Conservation and Recovery Act (RCRA) as D008 hazardous waste from off-site sources (USEPA, 2001a). This waste was converted into lead ingots using pot and rotary furnaces equipped with baghouses to collect particulate matter from the furnace that consisted predominantly of lead dust. The sludge that accumulated in the furnaces after smelting was classified as K069 waste hazardous waste. Finished lead ingots were stored in a roundhouse at the north end of the property prior to shipment off-site.

Based on background information, the by-products produced from smelting operations included furnace flux, slag, dross, baghouse fines and furnace sludge (USEPA, 2001a). With the exception of slag, which was tested and disposed of off-site, most of the lead-bearing by-products were recycled back into the furnace. Cooling water used in the operations was diverted to a combined sewer system operated by the NEORD (ESC, 1991).

Violations relating to noncompliance and poor operating practices are documented in various state and federal agency reports, summarized in the Section III of the AOC, presented in Appendix A of the RD/RA Workplan. In January of 1992, the OEPA installed three ambient air monitors near the facility property and quarterly air sampling from the station immediately downwind of the facility showed repeated exceedence of the Clean Air Act's 42 USC National Ambient Air Quality Standard (NAAQ) for lead. MMI installed a sprinkling system in July 1992 in an attempt to prevent air-borne migration of the dust from the facility (USEPA, 2001) but exceedences of the NAAQ for lead continued to be measured downwind of the facility. On September 9, 1992, MMI conducted a thorough cleaning of the facility in another attempt to minimize the effects of wind-blown facility dust.

On August 5, 1993, as a result of continuing RCRA violations, the Ohio EPA Director ordered MMI to cease operating the facility until it could demonstrate compliance (USEPA, 2001a). Operations never did resume at the MMI facility and Bank One of Ohio took possession of all MMI cash collateral and accounts receivable. The current property owner remains MMI. The

former facility president, Mr. Douglas Mickey, is deceased (USEPA, 2001).

Following shutdown, MMI and the USEPA continued negotiations to resolve RCRA noncompliance issues. On March 28, 1995 the USEPA RCRA Division deferred the MMI Site to CERCLA for cleanup. On August 22, 1995, MMI withdrew all permits still in effect regarding its operation terminating its ability to legally treat, store or dispose of hazardous waste at the facility (USEPA, 2001a). Fifty-three potentially responsible parties (PRP Respondent Group) signed an Administrative Order by Consent for the MMI facility that became effective April 17, 1997. The Order required the PRPs to conduct a Phase I TCR action and a Phase II Engineering Evaluation and Cost Analysis (EE/CA) for a non-time critical removal action for the facility pursuant to the National Contingency Plan (NCP) and the Superfund Accelerated Cleanup Model (SACM) guidance.

In accordance with the April 17, 1997 AOC Docket No: V-W-97-C both the Phase I TCR and Phase II EE/CA have been completed by ENTACT on behalf of the PRP Respondent Group, as described in Section 1.4 of this Workplan.

1.4 Description of Remedial Action

Based on the findings of the Phase II EE/CA, an AOC was entered into between the USEPA and the PRP Respondent Group in Spring 2002 to perform a non-critical removal action outlined in the Statement of Work (SOW) to address remaining lead impacts at the site that are associated with former facility operations.

The removal action includes the following tasks:

- Clear and grub areas requiring excavation of all trees and brush for disposal off-site.

- Demolish above-grade concrete and metal structures remaining on-site after the Phase I TCR demolition activities in accordance to the design specifications. Sized concrete construction debris will either be used as a sub-base material in areas to be covered with the asphalt cover or will be transported off-site disposal as construction debris. All wood, bricks or metal debris that are removed will be disposed of off-site as construction debris.

- Establish a coordinate grid system along the perimeter of the property outside the fence line and in on-property areas where excavation is required.

- Excavate off-property soils along the western, eastern and southern perimeter of the MMI facility, that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first. XRF screening technology will be used to guide the depth of the excavations during removal.

- Excavate designated on-property soils that are not under concrete or the proposed asphalt cover (including grids I1, J1 and K1 excavated during the Phase I TCR) that exceed the RBRG of 1,000 mg/Kg or until historic slag fill material is encountered, whichever comes first.

- Conduct confirmatory soil sampling from the excavation floor in grids where the excavation was terminated prior to reaching the historic slag fill material to confirm that all soils that are above the cleanup level have been excavated and removed.

- Backfill all excavated areas once verified to have met the RBRG or have reached historic slag fill, and grading to promote positive drainage in accordance with the design documents. Backfill for areas not covered by asphalt or concrete will be filled with clean imported fill material that has been approved for use based on analytical results and is suitable to maintain vegetative growth.

- Stabilize excavated soils to meet the applicable LDRs for contaminated soils for lead, and any underlying hazardous constituent (UHC) during waste profiling, to render the material nonhazardous for either use as fill in low areas beneath the proposed asphalt cover or for off-site disposal at an approved Subtitle D facility.

- Conduct verification sampling of treated soils using TCLP lead analysis to verify the material has been rendered non-hazardous for lead prior to either placement in low areas beneath the proposed asphalt cover or for off-site disposal as nonhazardous waste.

- Off-site disposal of all treated soils not used to fill low areas beneath the proposed asphalt cover, including stockpiled soils from the Holmden Properties Removal Action, in accordance with the SOW and the approved design plan.

- Place an asphalt cover over the deteriorated area of the concrete located in southern portion of the site in accordance with the design documents.

- Recondition existing concrete surfaces not under the asphalt cover by sealing any significant cracks and breaks that extend through the concrete surface, followed by encapsulation of the concrete surface, in accordance with the approved design plan.

- Abandon of all existing monitoring wells on site in accordance to applicable State of Ohio regulations (OAC-3745-9-10).

- Remove any existing solid waste including Investigative Derived Waste (IDW) from previous or current removal actions.

- Install a perimeter chain-link fence and three double-swing gates at the completion of the RA to control site access at the site in accordance with the design documents.

- Perform Operation and Maintenance activities to ensure the integrity of the remedy by maintaining and repairing the concrete and asphalt cover, and the perimeter fencing for a period of thirty (30) years, as required under CERCLA.

1.5 Highlights of Program

The community relations program for the Site is designed to allow the community to learn about, and participate in, the Superfund process.

The community relations program will include the following activities:

- A spokesperson for the Site will be designated to answer questions and concerns the community may have about the implementation of the RA.
- A spokesperson from the EPA will be designated to answer questions and concerns the community may have about the implementation of the RA.
- An information repository has been established so that the community has access to documents written in accordance with the AOC.
- The community will have the opportunity to review the major documents written for the AOC.

1.6 Documents Available for Public Review

Upon EPA approval of the final work plans and reports of findings required by the Consent Decree, the documents will be placed in a public location accessible to the community, i.e., public library. The workplan documents will be available for public review. A public notice will be published in the local newspaper to inform the community when documents require public notice or comment is required.

The documents that will be available for public review during the implementation of the RA are:

- The Removal Design/Removal Action Workplan
- The Field Sampling Plan
- The Quality Assurance Project Plan
- The Health and Safety Plan/Contingency Plan
- The Operation and Maintenance Plan
- The 1997 Treatability Study Report
- The Stormwater Control Plan
- The Pre-Design and Final Design Documents
- The Community Relations Plan

Reports that will be prepared and submitted during the course of this project include the following:

- Completion of Remedial Action Report
- Post-Excavation Confirmatory Soil Sampling Report
- Pre-final Inspection Report and Final Inspection Reports
- Monthly Reports during the implementation of the RA

1.7 Information Repositories

The workplan and reports required for the RA are available for review in the site information repository at the following location:

Jefferson Branch
Cleveland Public Library
850 Jefferson Avenue
Cleveland, Ohio
Phone: (216) 623-7004

1.8 List of Contacts

Questions or concerns related to the Remedial Design/Remedial Action Workplan being implemented at the Master Metals, Inc. Site in Cleveland, Ohio should be addressed to the following individuals:

Ms. Gwen Massenberg, Remedial Project Manager
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Mr. Michael Stoub, Project Coordinator
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